EPA/600/R-10/135
10/35/WQPC-SWP
September 2010

Environmental Technology Verification Report

Coatings for Wastewater Collection Systems

Protective Liner Systems, Inc.
Epoxy Mastic PLS-614

Prepared by

Center for Innovative Grouting Materials and Technology
University of Houston

For

NSF International

Under a Cooperative Agreement with

U.S. Environmental Protection Agency

ETV ETV ETV

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

U.S. Environmental Protection Agency **NSF International**

ETV Joint Verification Statement

TECHNOLOGY TYPE:	**Infrastructure Rehabilitation Technologies**	
APPLICATION:	**Coatings for Wastewater Collection Systems**	
TECHNOLOGY NAME:	**Protective Liner Systems Epoxy Mastic PLS-614 (PLS-614)**	
TEST LOCATION:	**University of Houston, CIGMAT**	
COMPANY:	**Protective Liner Systems, Inc.**	
ADDRESS:	**6691 Tribble Street**	**PHONE: (770) 482-5201**
	Lithonia, GA 30058	**FAX: (770) 484-1821**
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EPA created the ETV program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The program's goal is to further environmental protection by accelerating the acceptance and use of improved and more cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer-reviewed data on technology performance to those involved in the design, distribution, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations; stakeholder groups, which consist of buyers, vendor organizations, and permitters; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

NSF International (NSF), in cooperation with the U.S. Environmental Protection Agency (EPA), operates the Water Quality Protection Center (WQPC), one of six centers under the Environmental Technology Verification (ETV) Program. The WQPC recently evaluated the performance of the Protective Liner Systems PLS-614 epoxy mastic, marketed by Protective Liner Systems, Inc. The PLS-614 coating was tested at the University of Houston's Center for Innovative Grouting Materials and Technology (CIGMAT).

TECHNOLOGY DESCRIPTION

The following description of the Protective Liner Systems coating material (PLS-614) was provided by the vendor and does not represent verified information.

Protective Liner Systems' PLS-614 is a 100% solids epoxy coating used for structural concrete protection, rehabilitation and repair, and is designed to be applied by trowel or spray. The PLS-614 system is formulated to provide a monolithic structural coating or patch for rehabilitation of concrete structures and protection against wear, corrosion, infiltration and exfiltration.

VERIFICATION TESTING DESCRIPTION - METHODS AND PROCEDURES

The objective of this testing was to evaluate PLS-614 used in wastewater collection systems to control the deterioration of concrete and clay infrastructure materials. Specific testing objectives were (1) to evaluate the acid resistance of PLS-614 coated concrete specimens and clay bricks, both with and without holidays (small holes intentionally drilled through the coating and into the specimens to evaluate chemical resistance), and (2) determine the bonding strength of PLS-614 to concrete and clay bricks.

Verification testing was conducted using relevant American Society for Testing and Materials (ASTM)[1] and CIGMAT[2] standards, as described below. Product characterization tests were conducted on the coating material and the uncoated concrete and clay specimens to assure uniformity prior to their use in the acid resistance and bonding strength tests. Protective Liner Systems' representatives were responsible for coating the concrete and clay specimens, under the guidance of CIGMAT staff members. The coated specimens were evaluated over the course of six months.

PERFORMANCE VERIFICATION

(a) Holiday Test - Chemical Resistance

PLS-614 coated concrete cylinders and clay bricks were tested with and without holidays (small holes intentionally drilled through the coating) in deionized (DI) water and a 1% sulfuric acid solution (pH=1). A total of 20 coated concrete specimens and 20 coated clay brick specimens were exposed. Specimens were cured for two weeks prior to creation of 0.12 in. and 0.50 in. holidays. The 0.12 in. holidays were exposed to both DI water and acid solution, while the 0.50 in. holidays were exposed only to the acid solution. Observation of the specimens at 30 and 180 days was made for changes in appearance such as blistering or cracks in the coating around the holiday or color changes in the coating. Control tests were also performed using specimens with no holidays. A summary of the chemical exposure observations is presented in Table 1.

Table 1. Summary of Chemical Exposure Observations

Specimen Material (Coating Condition)	DI Water (days)				1% H_2SO_4 Solution (days)				Comments
	Without Holidays		With Holidays		Without Holidays		With Holidays		
	30	180	30	180	30	180	30	180	
Concrete – Dry	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Color change in coating submerged in acid solution.
Concrete – Wet	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Color change in coating submerged in acid solution.
Clay Brick – Dry	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Color change in coating submerged in acid solution.
Clay Brick – Wet	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Color change in coating submerged in acid solution.

N = No blister or crack; (n) = Number of specimens.

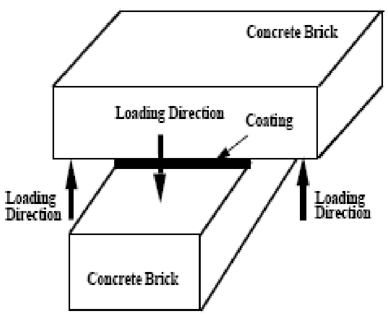
A specimen made only of PLS-614 was submerged in water for 10 days, showing no weight change over the period. Over an exposure time of 180 days, coated concrete specimens with no holidays showed less than 0.7% gain in DI water and acid exposures, as did clay brick specimens exposed to DI water. Coated clay brick exposed to acid showed a 2-7% weight gain. With holidays, coated concrete specimens showed up to 1.2% weight change, while coated clay brick specimens showed 5-7% gains. Changes in the diameters/dimensions of the specimens at the holiday levels were negligible after 180 days of exposure.

(b) Bonding Strength Tests (Sandwich Method and Pull-Off Method)

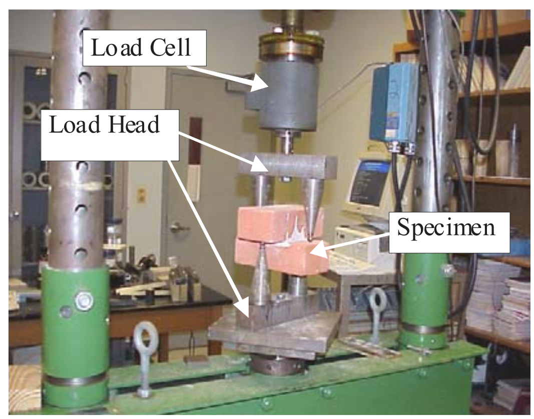
Bonding strength tests were performed to determine the bonding strength between the PLS-614 coating and concrete/clay brick specimens over a period of six months. Eight sandwich (4 dry-condition, 4 wet-condition) and sixteen pull-off (8 dry-condition, 8 wet-condition) tests were performed on both coated concrete samples and coated clay bricks.

Sandwich Test Method (CIGMAT CT 3)

CIGMAT CT 3, a modification of ASTM C321-94, was used for the testing. PLS-614 was applied to form a sandwich between a like pair of rectangular specimens (Figure 1 (a)), both concrete brick and clay brick, and then tested for bonding strength and failure type following a curing period. The bonding strength of the coating was determined using a load frame (Figure 1 (b)) to determine the failure load and bonding strength (the failure load divided by the bonded area). The sandwich bonding tests were completed at 30, 90 and 180 days after application of the PLS-614.



(a) Test specimen configuration



(b) Load frame test setup

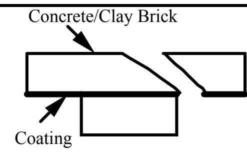
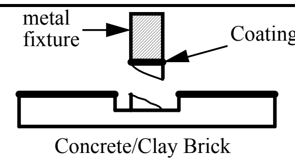
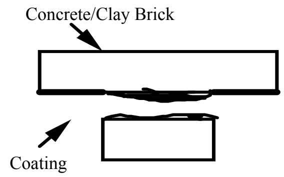
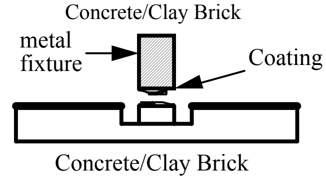
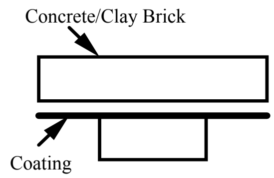
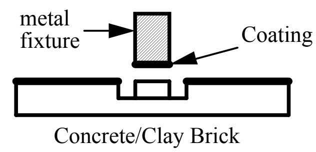
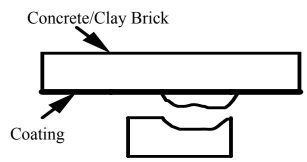
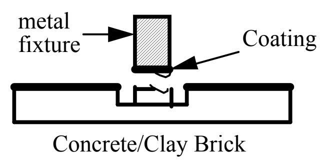
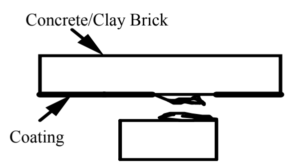
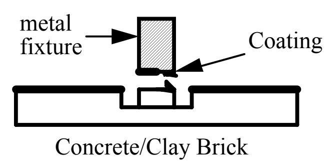
Figure 1. Bonding test arrangement for sandwich test.

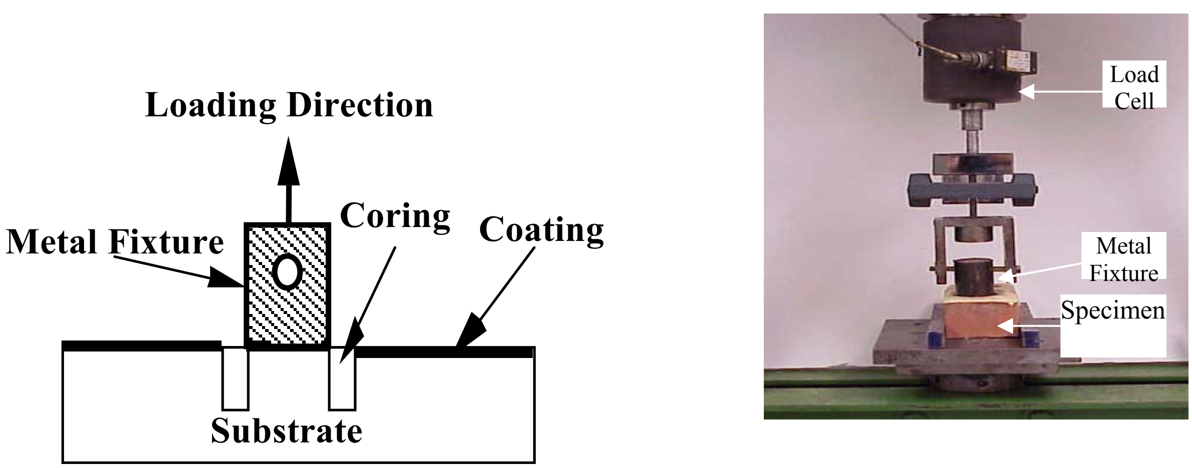
Dry-coated specimens were dried at room conditions for at least seven days before they were coated, while wet-coated specimens were immersed in water for at least seven days before the specimens were coated. Bonded specimens were cured under water up to the point of testing. The type of failure was also characterized during the load testing, as described in Table 2.

Pull-Off Method (CIGMAT CT 2)

Per CIGMAT CT 2, a 2-in. diameter circle was cut into coated concrete prisms and clay bricks to a predetermined depth to isolate the coating, and a metal fixture was glued to the isolated coating section using a rapid setting epoxy. Testing was completed on a load frame with the arrangements shown in Figure 2, with observation of the type of failure, as indicated in Table 2. The specimens were prepared in the same manner as for the sandwich test. The specimens were stored under water in plastic containers and the coatings were cored 24 hrs prior to the testing. The bonding tests were completed at 21, 60 and 180 days after application of the PLS-614. Results of the bonding tests are included in Table 3.

Table 2. Failure Types in Sandwich and Pull-Off Tests

Failure Type	Description	Sandwich Test	Pull-off Test
Type-1	Substrate Failure		
Type-2	Coating Failure		
Type-3	Bonding Failure		
Type-4	Bonding and Substrate Failure		
Type-5	Bonding and Coating Failure		



(a) Specimen preparation **(b) Load frame arrangement**

Figure 2. Pull-off test method load frame arrangement.

Table 3. Summary of Test Results for Bonding Strength Tests (12 Specimens for Each Condition)

Substrate – Application Condition	Test [1]	Failure Type [2] – Number of Failures					Failure Strength (psi)	
		1	2	3	4	5	Range	Average
Concrete – Dry	Sandwich	4					232 – 293	269
	Pull-off	1			7		107 – 304	205
Concrete – Wet	Sandwich	3			1		257 – 321	287
	Pull-off	5			3		190 – 350	234
Clay Brick – Dry	Sandwich	4					314 – 350	335
	Pull-off	3			5		187 – 321	253
Clay Brick – Wet	Sandwich	4					338 – 384	366
	Pull-off	3			5		181 – 374	264

[1]Sandwich test (CIGMAT CT-2/Modified ASTM D 4541-85) or Pull-off test (CIGMAT CT-3/ASTM C 321-94).
[2]See Table 2.

(c) Summary of Verification Results

The performance of the Protective Liner System, Inc. PLS-614 epoxy mastic for use in wastewater collection systems was evaluated for chemical resistance and the bond of the coating with both wet and dry substrate materials, made up of concrete and clay brick. The type of bonding test, whether sandwich test or pull-off test, impacted the mode of failure and bonding strength for both substrate materials. The testing indicated:

General Observations

- Samples of the coating material alone showed no weight gain when exposed to water over a 10-day period.
- None of the coated concrete or clay brick specimens, with or without holidays, showed any indication of blisters or cracking during the six-month holiday-chemical resistance tests.
- There were no observed changes in the dimensions of the coated concrete or clay brick specimens at the holiday levels for either DI or acid exposures.
- All 48 of the bonding tests resulted in substrate and substrate/bonding failures, with 27 substrate failures (Type-1) and 21 bonding/substrate failures (Type-4).

Concrete Substrate

- Weight gain was < 0.60% for any of the coated concrete specimens without holidays.
- Weight gain was < 1.5% for any of the coated specimens with holidays for both water and acid exposures.
- Dry-coated concrete failures were mostly (7 of 12) bonding and concrete substrate (Type -4) failures, with the remainder being concrete substrate (Type-1) failures.
- Average tensile bonding strength for dry-coated concrete specimens was 226 psi, with individual specimens ranging from 107 to 304 psi.
- Wet-coated concrete failures were mostly (8 of 12) concrete substrate (Type-1) failures, with the remainder being bonding and concrete substrate (Type-4) failures.
- Average tensile bonding strength for wet-coated concrete specimens was 252 psi, with individual specimens ranging from 190 to 350 psi.

Clay Brick Substrate

- Without holidays, weight gain was < 0.45% for water exposed coated clay brick specimens; weight gain for acid exposed coated clay brick specimens was about 2-7%.
- With holidays, weight gains were > 5% for water exposed specimens and generally > 6% for acid exposed specimens; the holiday size did not make a significant difference in weight gain.
- Dry-coated clay brick failures were mostly (7 of 12) clay brick substrate (Type -1) failures, with the remaining five being bonding and clay brick substrate (Type-4) failures.
- Average tensile bonding strength for dry-coated clay brick specimens was 280 psi, with individual specimens ranging from 187 to 350 psi.
- Wet-coated clay brick failures were predominantly (7 of 12) clay brick substrate (Type-1) failures, with the remaining five being bonding and clay brick substrate (Type-4) failures.
- Average tensile bonding strength with-wet coated clay brick was 286 psi, with individual specimens ranging from 181 to 384 psi.

Quality Assurance/Quality Control

NSF completed a technical systems audit prior to the start of testing to ensure that CIGMAT was equipped to comply with the test plan. NSF also completed a data quality audit of at least 10% of the test data to ensure that the reported data represented the data generated during testing.

Original signed by *Sally Gutierrez*	*October 6, 2010*	*Original signed by* *Robert Ferguson*	*November 2, 2010*
Sally Gutierrez Director National Risk Management Research Laboratory Office of Research and Development United States Environmental Protection Agency	Date	Robert Ferguson Vice President Water Systems NSF International	Date

Availability of Supporting Documents

Referenced Documents:

1) Annual Book of ASTM Standards (1995), Vol. 06.01, Paints-Tests for Formulated Products and Applied Coatings, ASTM, Philadelphia, PA.
2) CIGMAT Laboratory Methods for Evaluating Coating Materials, available from the University of Houston, Center for Innovative Grouting Materials and Technology, Houston, TX.

Copies of the *Test Plan for Verification of Protective Liner Systems PLS-614 Coating for Wastewater Collection Systems* (March 2009), the verification statement, and the verification report (NSF Report Number 10/34/WQPC-SWP) are available from:

ETV Water Quality Protection Center Program Manager (hard copy)
NSF International
P.O. Box 130140
Ann Arbor, Michigan 48113-0140

NSF website: http://www.nsf.org/etv (electronic copy)
EPA website: http://www.epa.gov/etv (electronic copy)

Environmental Technology Verification Report

Verification of Coatings for Rehabilitation of Wastewater Collection Systems

Protective Liner Systems, Inc.

Prepared by

Center for Innovative Grouting Materials and Technology (CIGMAT)
University of Houston
Houston, TX 77204

Prepared for

NSF International
Ann Arbor, MI 48105

Under a cooperative agreement with the U.S. Environmental Protection Agency

Raymond Frederick, Project Officer
ETV Water Quality Protection Center
Water Supply and Water Resources Division
National Risk Management Research Laboratory
U.S. Environmental Protection Agency
Edison, New Jersey 08837

September 2010

NOTICE

The U.S. Environmental Protection Agency (USEPA) through its Office of Research and Development has financially supported and collaborated with NSF International (NSF) under a Cooperative Agreement. The Water Quality Protection Center, Source Water Protection area, operating under the Environmental Technology Verification (ETV) Program, supported this verification effort. This document has been peer reviewed and reviewed by NSF and USEPA and recommended for public release.

FOREWORD

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threaten human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites, sediments and ground water; prevention and control of indoor air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by: developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

TABLE OF CONTENTS

FIGURES

TABLES

ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
CIGMAT	Center for Innovative Grouting Materials and Technology, University of Houston
°C	Celsius degrees
°F	Fahrenheit degrees
DI	Deionized (water)
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
ft/sec or fps	Feet per second
ft^2	Square foot (feet)
gal	Gallons
holiday	A gap or void in the coating
hr	Hour(s)
in.	Inch(es)
kg	Kilogram(s)
L	Liter
lbs	Pounds
NRMRL	National Risk Management Research Laboratory
m^3	Cubic meters
mg/L	Milligram(s) per liter
mL	Milliliter(s)
mm	Millimeter(s)
MPa	MegaPascal(s)
NSF	NSF International
pcf	Pounds per cubic foot
psi	Pounds per square inch
QA	Quality assurance
QC	Quality control
Room conditions	23ºC ±2ºC and relative humidity of 50% ±5%
TO	Testing Organization
VO	Verification Organization (NSF)
VTP	Verification Test Plan
WQPC	Water Quality Protection Center

SECTION 1
INTRODUCTION

1.1 ETV Purpose and Program Operation

The U.S. EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The ETV Program's goal is to further environmental protection by substantially accelerating the acceptance and use of innovative, improved and more cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer reviewed data on technology performance to those involved in the design, distribution, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations (TOs); stakeholders groups that consist of buyers, vendor organizations, consulting engineers, and regulators; and the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

In cooperation with EPA, NSF operates the Water Quality Protection Center (WQPC), one of six centers under ETV. The WQPC developed verification testing protocols and generic test plans that serve as templates for conducting verification tests for various technologies. Verification of the Protective Liner Systems, Inc. Epoxy Mastic PLS-614 was completed following the Generic Test Plan for Verification of Coatings for Wastewater Collection Systems, 2008. The Generic Plan was used to develop a product-specific test plan for the PLS-614 coating.

1.2 Roles and Responsibilities

The ETV testing of Protective Liner Systems coating was a cooperative effort between the following participants:

- NSF
- US EPA
- University of Houston - CIGMAT
- Protective Liner Systems, Inc.

1.2.1 Verification Organization (NSF)

- Coordinate with CIGMAT, the TO, and the vendor to prepare and approve a product-specific test plan using this generic test plan as a template and meeting all testing requirements included herein;

- Coordinate with the ETV Coatings Technical Panel, as needed, to review the product-specific test plan prior to the initiation of verification testing;
- Coordinate with the EPA WQPC Project Officer to approve the product-specific verification test plan (VTP) prior to the initiation of verification testing;
- Review the quality systems of the testing organization and subsequently, qualify the TO;
- Oversee the coatings evaluations and associated laboratory testing;
- Review data generated during verification testing;
- Oversee the development of a verification report and verification statement;
- Print and distribute the verification report and verification statement; and
- Provide quality assurance oversight at all stages of the verification process.

Primary contact: Mr. Thomas Stevens
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789 North Dixboro Road
Ann Arbor, MI 48105
Phone: 734-769-5347
Email: stevenst@nsf.org

1.2.2 U.S. Environmental Protection Agency (EPA)

This verification report has been developed with financial and quality assurance assistance from the ETV Program, which is overseen by the EPA's Office of Research and Development (ORD). The ETV Program's Quality Assurance Manager and the WQPC Project Officer provided administrative, technical, and quality assurance guidance and oversight on all ETV WQPC activities. The primary responsibilities of EPA personnel were to:

- Review and approve VTPs, including the quality assurance project plans (QAPPs);
- Sign the VTP signoff sheet;
- Review and approve the verification report and verification statement; and
- Post the verification report and verification statement on the EPA ETV website.

Primary contact: Mr. Ray Frederick
Project Officer, Water Quality Protection Center
U.S. Environmental Protection Agency, NRMRL
2890 Woodbridge Ave. (MS-104)
Edison, New Jersey 08837
Phone: 732-321-6627
Email: frederick.ray@epamail.epa.gov

1.2.3 Testing Organization (CIGMAT Laboratories at UH)

The TO for this verification was CIGMAT Laboratories at the University of Houston. The primary responsibilities of the TO were:

- Coordinate with the VO and vendor relative to preparing and finalizing the product-specific VTP;

- Sign the VTP signoff sheet;
- Conduct the technology verification in accordance with the VTP, with oversight by the VO;
- Analyze all samples collected during the technology verification process, in accordance with the procedures outlined in the VTP and referenced SOPs;
- Coordinate with and report to the VO during the technology verification process;
- Provide analytical results of the technology verification to the VO; and
- If necessary, document changes in plans for testing and analysis, and notify the VO of any and all such changes before changes are executed.

CIGMAT supports faculty, research fellows, research assistants and technicians. The CIGMAT personnel worked in groups to complete the tests described in this report. All personnel report to the Group Leader and the CIGMAT Director. The CIGMAT Director is responsible for appointing Group Leaders, who, with his approval, are responsible for drawing up the schedule for testing. Additionally, a Quality Assurance (QA) Engineer, who is independent of the testing program, was responsible for internal audits.

Primary contact: Dr. C. Vipulanandan
University of Houston, CIGMAT
4800 Calhoun
Houston, Texas 77004
Phone: 713-743-4278
Email: cvipulanandan@uh.edu

1.2.4 Vendor (Protective Liner Systems, Inc.)

- Complete a product data sheet prior to testing (refer to Appendix D);
- Provide the TO with coating samples for verification (this includes applying the coating materials to test specimens at the CIGMAT facilities);
- Sign the VTP signoff sheet;
- Provide start-up services and technical support as required during the period prior to the evaluation;
- Provide technical assistance to the TO during verification testing period as requested; and
- Provide funding for verification testing.

Primary contact: Mr. Joseph Trevino
Protective Liner Systems, Inc.
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Email: www.protectivelinersystems.com

1.2.5 Technology Panel

A technology panel was formed to assist with the review of the generic coatings test plan. Input from the panel ensured that data generated during verification testing were relevant and that the method of evaluating different technologies was fair and consistent. The product-specific VTP was reviewed by representatives of the technology panel and approved by the WQPC Program Manager, the WQPC Project Officer, and the vendor.

1.3 Background and Technical Approach

University of Houston (UH)/CIGMAT researchers have been investigating the performance of various coatings for use in the City of Houston's wastewater facilities. Performance of each coating has been studied with wet (representing rehabilitation of existing wastewater collection systems) and dry (representing new construction) concrete and clay bricks. The studies have focused on:

- Applicability and performance of the coating under hydrostatic pressure (with an evaluation period between six to nine months);
- Chemical exposure with and without holidays (a gap or void in the coating) in the coating (initial evaluation period of six months); and
- Bonding strength (initial evaluation period of twelve months).

Chemical tests and bonding tests on over twenty coating materials are being continued at UH. The long-term data collected on each coating will help engineers and owners to better understand the durability of coated materials in wastewater environments. The overall objective of this testing program is to systematically evaluate coating materials used in wastewater systems to control the deterioration of cementitious materials.

Testing used relevant ASTM and CIGMAT standards. Specimens made from the coating material, in addition to uncoated concrete and clay specimens, first undergo characterization testing to determine their suitability for use during acid resistance and bonding strength tests. The coating manufacturer then coats the concrete and clay specimens, under the guidance of CIGMAT staff members. The coated specimens are then evaluated over the course of six months.

1.4 Objectives

The objective of this ETV study was to evaluate the Protective Liner Systems International Inc. Protective Liner Systems Epoxy Mastic PLS-614 (PLS-614) (dry and wet) for use in sewer rehabilitation projects. Specific objectives are to:

- Evaluate the acid resistance of the coated concrete and clay bricks with and without holidays; and
- Determine the bonding strength of the coating materials to concrete and clay bricks over a period of time.

A coating-specific VTP was prepared for the Protective Liner Systems coating material. The VTP included specific testing procedures and a quality assurance project plan (QAPP) describing the quality systems to be used during the evaluation.

1.5 Test Facility

The testing was performed in the CIGMAT Laboratories at the University of Houston, Houston, Texas. The CIGMAT Laboratories and affiliated facilities are equipped with devices that can perform all of the coatings tests. Molds are available to prepare the specimens for testing, and all acid resistance and bonding strength test procedures are documented in standard operating procedures.

SECTION 2
COATING DESCRIPTION

The coating material evaluated in this verification was the Protective Liner Systems International Inc. Protective Liner Systems Epoxy Mastic PLS-614 (PLS-614). The Vendor Data Sheet characterizing the coating material is included in Appendix D. The coating is described on the Protective Liner Systems International Inc. web site (http://www.protectivelinersystems.com) as a concrete polymer paste used for structural concrete protection, rehabilitation and repair. Protective Liner Systems' PLS-614 is a 100% solid epoxy, designed to be applied by trowel. The PLS-614 system is formulated to provide a coating, or patch for rehabilitation of concrete and protection against corrosion.

The application instructions for the PLS-614 were:

> Apply a maximum of 100 to 150 mils of the coating to protect concrete and clay bricks. No primer is used. The curing time for the coating is 6 hours. The coating is applied using a trowel.

The key to successful coating is preparation of the surface to be coated. Per Protector Liner System's web site, preparation for application of their coating requires:

> Use high-pressure water washing to prepare the surface.

The coating is gray in color, as shown in Figure 2-1 for a pure coating sample. Photos of the applied coating at the time of bonding tests are provided in Section 4.

Figure 2-1. Specimen of pure Protective Liner Systems PLS-614.

SECTION 3
METHODS AND TEST PROCEDURES

The Verification Test Plan (VTP) includes a detailed description of the testing completed for the Protective Liner Systems PLS-614. The testing involved characterization of the coating material, as well as holiday tests and bonding strength tests on the coated/lined specimens. The following is a summary of the methods and test procedures used in this verification.

3.1 Preparation of Test Specimens

Testing was completed using both concrete and clay brick specimens prepared in the CIGMAT Laboratory by CIGMAT personnel prior to application of the coating. Concrete specimens were created by CIGMAT staff, while standard sewer clay bricks were obtained from a local brick supplier and the specimens prepared to the proper specifications by CIGMAT staff.

3.1.1 Preparation of the Concrete Specimens

Cylindrical and prism specimens were used during testing. Mix proportions for the concrete are summarized in Table 3-1. The cylindrical specimens were cast in 3-in. (diameter) × 6-in. (length) plastic molds, while wooden molds were used to cast the approximately 2.25-in. × 3.75-in. × 8-in. prism specimens.

Table 3-1. Mix Proportions for Concrete Specimens

Materials	Amount	Specification
Cement	6 bags	ASTM C150 Type 1 (purchased in 94 lb bags)
Sand	1400 -1500 lbs	ASTM C33
Coarse Aggregate	1600 -1700 lbs	ASTM C33 (ranging in size from No. 4 to 1.5 in. sieve)
Water	320 – 330 lbs	Tap water

Proper proportions of cement, sand, coarse aggregate and water were mixed in a concrete mixer until uniform in appearance, and the molds were filled with the mixture and mechanically vibrated to the appropriate consistency. The specimens were cured for at least 28 days at room conditions (23ºC ± 2ºC and relative humidity of 50% ± 5%).

3.1.2 Preparation of Clay Brick Specimens

Standard sewer clay bricks used for the chemical exposure testing (holiday test) were cut approximately in half using a diamond-tipped saw blade at the CIGMAT Laboratory, resulting in approximately 1.5-in. × 3.75-in. × 6-in. prism specimens. The prepared specimens were stored at room conditions until use. Bonding tests were completed using whole clay bricks.

3.1.3 Coating Specimens

Specimens made of the Protective Liner Systems PLS-614 only were also prepared in 1.5-in. (diameter) × 3-in. (length) plastic molds. As indicated in Section 3.2, these specimens were analyzed and are reported to provide basic data that will be available to verify that the coating used in any future application is the same as applied for this verification testing.

3.2 Evaluation of Specimens

The concrete cylinders and prisms, clay brick prisms, and raw coating material cylinders were evaluated to determine their properties under the described test conditions. The specimens were characterized using the tests shown in Table 3-2.

Table 3-2. Test Names / Methods

Test Name	Test Method
Pulse Velocity	ASTM C 597
Holiday Test (Chemical Resistance)	ASTM G20 / CIGMAT CT-1-99
Bonding Strength	ASTM C 321/ CIGMAT CT-3 (Sandwich Method) ASTM D 4541/CIGMAT CT-2 (Pull-Off Strength)

The pulse velocity and unit weight of all the specimens were determined for quality control purposes. Additional specimens were used to determine the compressive (3 specimens) and flexural strength (3 specimens) of concrete and flexural strength of clay bricks (3 specimens) (Table 3-3). Note that the strength tests were done for completeness and not for quality control.

Table 3-3. Number of Specimens Used for Each Characterization Test

Material	Number of Specimens Used in Test				
	Unit weight	Pulse velocity [1]	Water absorption [2]	Flexure [3]	Compression [3]
Coating	6	6	6	N/A	N/A
Concrete Cylinders	20	20	10	N/A	3
Concrete Prisms	36	36	N/A	3	N/A
Clay Prisms	56	56	10	3	N/A

[1] Unit weight measurement taken on specimens prior to this test.
[2] Specimens used after the Pulse Velocity test.
[3] Flexure and compression tests are performed for informational purposes only.

3.3 Coating Application

The concrete and clay specimens were coated by a representative of Protective Liner Systems Inc. in the CIGMAT Laboratory at the University of Houston, in the presence of CIGMAT staff. Wet specimens were immersed in water for at least seven days before coating the specimens. All test specimens for the laboratory tests were prepared at the University of Houston Test Site. Prior to applying the coating, the surfaces of the specimens were sand blasted. The coating was applied directly to the specimen surfaces by trowel, with no primer prior to application. Protective Liner recommends, in actual use, an application of 125 mil thickness. Per Protective Liner Systems, the finished coating thickness was approximately 100 to 150 mils thick. This thickness was not verified by the TO, as the thickness of the applied coating does not impact the testing. The application temperature was 72º F and humidity was typical of room conditions. Protective Liner indicates a cure time of approximately four hours (at 70º F).

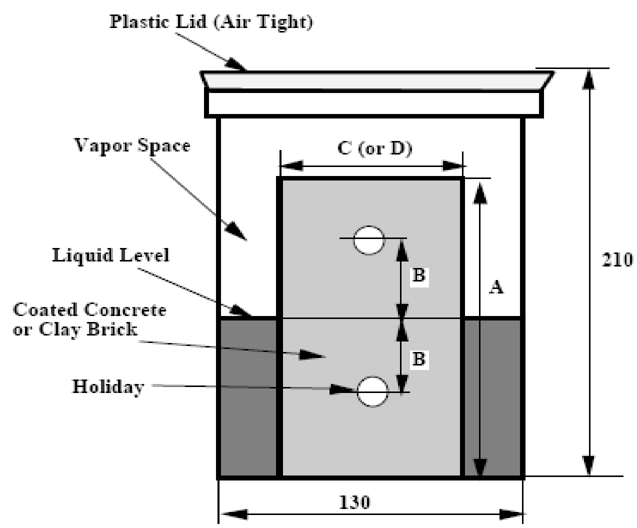
3.4 Evaluation of Coated Specimens

3.4.1 Holiday Test (CIGMAT CT-1)

The holiday test (CIGMAT CT-1, a modification of ASTM G20-88 used with concrete and clay brick materials) is a relatively rapid test to evaluate the acid resistance of coated concrete and clay brick specimens under anticipated service conditions. The test provides information about changes occurring to the specimens under two reagent conditions: (1) deionized (DI) water (pH = 5 to 6) and (2) 1% sulfuric acid solution (a pH of 1), which represents a long-term, worst-case condition in a wastewater collection system, arising from formation of hydrogen sulfide.

Changes in the specimens were monitored at regular intervals, including (1) diameter/dimension at the holiday level, (2) weight of the specimen, and (3) physical appearance of specimen. Control tests were also performed using specimens with no holidays.

Both wet and dry specimens were coated on all sides. As shown in Figure 3-1, two radial holidays of different diameters were drilled along the same axis into each specimen to a depth of approximately 1/2 in. The holiday diameters used during this test were 1/8 in. and 1/2 in. Specimens were cured for approximately 15 days prior to drilling the holes. This provided time to be sure the coating had sufficiently cured prior to the creation of the holidays so the physical action of the drill bits would not impact the integrity of the bond between the coating and the substrate at the location of the holiday. Half the specimen was submerged in the test liquid and half remained in the vapor space above the liquid. The specimens were stored at room temperature (72^{o}F). The typical cure time for the coating is six hours.



A ----- 152 mm (6.0 in.) height concrete specimen or clay brick
B ----- 38 mm (1.5 in.) holiday location
C ----- 76 mm (3 in.) diameter concrete cylinder
D ----- 152 x 64 x 45 mm cross section of clay brick

Figure 3-1. Test configuration for the holiday test.

The specimens were inspected after one and six months to determine if there were blisters, cracking of the coating, and/or erosion of the coating arising from the exposure. At the time of the inspections, the coated specimens were given ratings shown in Table 3-4.

Table 3-4. Ratings for Chemical Resistance Test Observations

Rating	Rating Notation	Observation
No significant change	N	No visible blister; no cracking.
Blister	B	Visible blister up to one inch in diameter; no cracking.
Cracking	C	Blister with diameter greater than one inch and/or cracking of coating at the holiday.

Further information regarding the chemical resistance testing, including a description of the coating failure mechanisms may be found at the following web site:

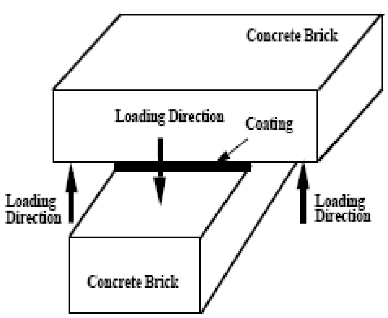
http://cigmat.cive.uh.edu/content/conf_exhib/99_poster/2.htm

3.4.2 Bonding Strength Tests (Sandwich Method and Pull-Off Method)

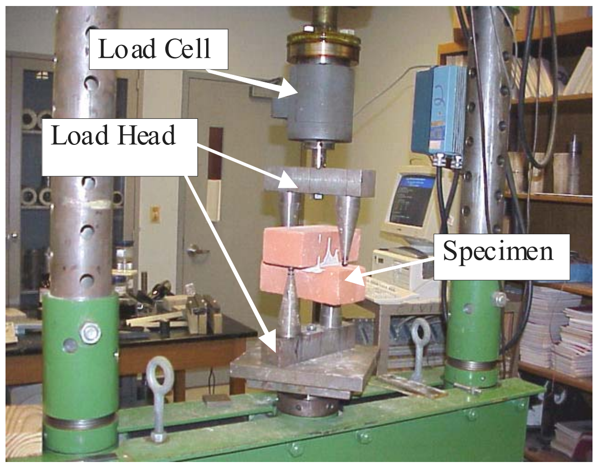
These tests are performed to determine the bonding strength between concrete/clay brick specimens and the coating material over a period of six months. Eight sandwich and twelve pull-off tests, for both dry and wet conditions, were performed on both coated concrete samples and coated clay bricks.

3.4.2.1 Sandwich Test Method (CIGMAT CT-3)

For this test (CIGMAT CT-3, a modification of ASTM C321-94), the coating was applied to form a sandwich between a like pair of rectangular specimens (Figure 3-2 (a)), both concrete prisms and clay brick, and then tested for bonding strength and failure type following a curing period. The bonding strength of the coating was determined using a load frame (Figure 3-3 (b)) to determine the axial failure load, which is divided by the bonded area to determine the bonding strength.



(a) Test specimen configuration



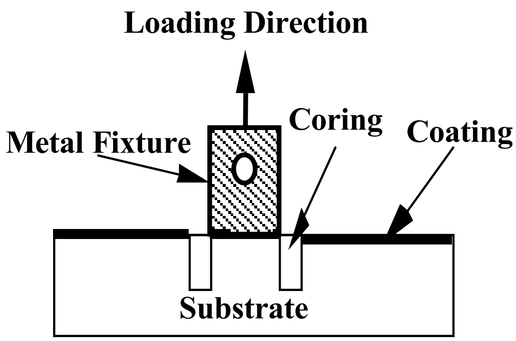
(b) Load frame test setup

Figure 3-2. Bonding test arrangement for sandwich test.

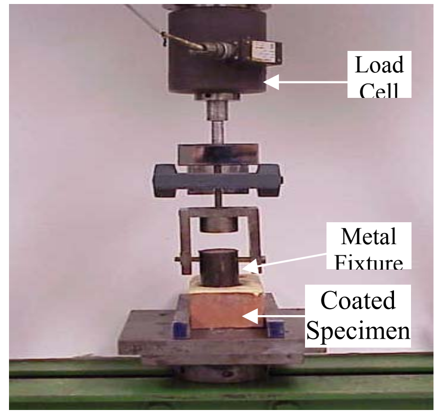
Both dry and wet specimens were used to represent extreme coating conditions. Dry specimens were dried at room conditions for at least seven days before they were coated, while wet specimens were immersed in water for at least seven days before the specimens were coated. Bonded specimens were cured under water up to the point of testing. At the same time as the load testing, the type of failure was also characterized, as described in Table 3-5.

3.4.2.2 Pull-Off Method (CIGMAT CT-2)

For this test (CIGMAT CT-2), a 2-in. diameter circle was cut into coated concrete prisms and clay bricks to a predetermined depth to isolate the coating, and a metal fixture was glued to the isolated coating section using a rapid setting epoxy. Testing was completed on a load frame with the arrangements shown in Figure 3-3, with observation of the type of failure, as indicated in Table 3-5. The specimens were prepared in the same manner as for the sandwich test. The specimens were stored under water in plastic containers and the coatings were cored 24 hrs prior to the test.



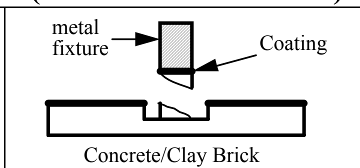
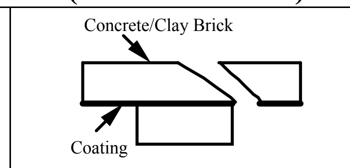
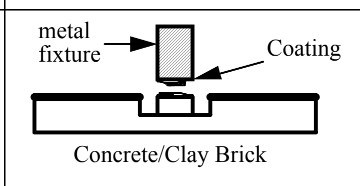
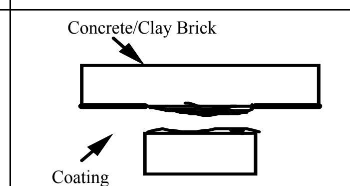
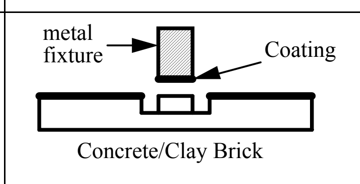
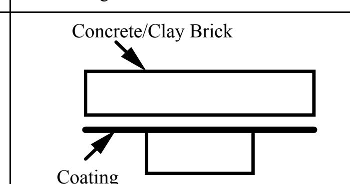
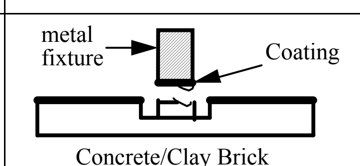
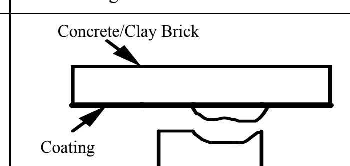
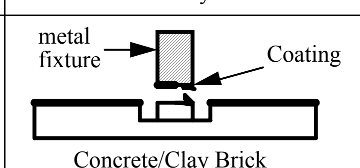
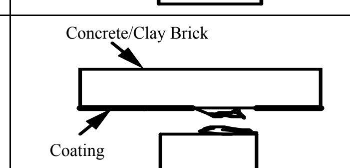
(a) Specimen preparation



(b) Load frame arrangement

Figure 3-3. Pull-off test method load frame arrangement.

Table 3-5. Failure Types in Pull-Off and Sandwich Tests

Failure Type	Description	CIGMAT CT 2 Test (Modified ASTM D4541)	CIGMAT CT 3 (ASTM C321 Test)
Type-1	Substrate Failure		
Type-2	Coating Failure		
Type-3	Bonding Failure		
Type-4	Bonding and Substrate Failure		
Type-5	Bonding and Coating Failure		

Type-1 failure is substrate failure. This is the most desirable result if the bonding strength is quite high (in the range 8% to 12% of the concrete substrate compressive strength). In Type-2 failure, the coating has failed. Type-3 failure is bonding failure where failure occurred between the coating and substrate. Type-4 and Type-5 are combined failures. Type-4 failure is the bonding and substrate failure where the failure occurs in the substrate and on the interface of the coating and the substrate. This indicates that the adhesive strength is comparable with the tensile strength of substrate. Type-5 failure is coating and bonding failure where the failure occurs due to low cohesive and adhesive strength of the coating.

3.5 Testing Events

The frequency of testing events is summarized in Table 3-6. The timing of the coated sample testing was spaced so data would be obtained during an initial period (within the first 30 days), an intermediate period (three months) and long period (six months). It is not critical that the testing be completed at exactly 30 days, 90 days or 180 days, as the measurements provide an indication of any change in coating bonding over the six month period.

Table 3-6. Test Frequency

Approximate Exposure Times	Holiday Test*		Bonding Strength Test	
	DI Water	1% H_2SO_4	Sandwich	Pull-Off
30 days	20	20	8	16
90 days			4	8
180 days	20	20	4	8

* The same specimens are monitored for 6 months.

SECTION 4
RESULTS AND DISCUSSION

The testing was designed to evaluate the ability of the Protective Liner Systems PLS-614 coating (coating) to adhere to a substrate under varying conditions. Dry coating condition simulates a new concrete surface while wet condition simulates a rehabilitation condition. Adhesion was evaluated by three methods – introducing holidays in coated specimens to determine if exposure of the substrate to corrosive conditions impacts the bond of the coating to the substrate, determining the bond strength of the coating between two substrates and determining the bond strength of the coating to a single substrate.

4.1 Test Results

4.1.1 Coating Specimens

Six pure specimens of the coating were evaluated for unit weight, pulse velocity and water absorption to provide basic data that will be available to verify that the coating used in any future application is the same as applied for this verification testing. The specimens were immersed in water for 10 days and showed no weight gain over the time frame. The unit weight varied from 87 pcf to 91 pcf, with an average of 89 pcf and a coefficient of variation of 1.8%. The pulse velocity varied from about 8020 fps to about 8230 fps, averaging about 8130 fps with a standard deviation of 72 and a coefficient of variation of 0.9%. All data is provided in Table 4-1.

Table 4-1. Properties of Coating Samples (Protective Liner Systems PLS-614)

Specimen	Unit Weight (pcf)	Pulse Velocity (fps)
1	90.2	8104
2	86.8	8229
3	90.9	8171
4	87.5	8149
5	88.7	8016
6	88.3	8131
Average	88.7	8134
Standard Deviation	1.57	71.3
Coefficient of Variation (COV)	1.8%	0.9%

4.1.2 Coated Materials

As stated in previous sections, the evaluation of the coating was accomplished in two phases – chemical resistance and bonding strength.

4.1.2.1 Holiday Test - Chemical Resistance

In order to evaluate the performance of PLS-614, coated concrete cylinders and clay bricks were tested with and without holidays in DI water and a 1% sulfuric acid solution (pH=1). Performance of PLS-614 was evaluated over a period of six months, from February 2009 to August 2009, with monthly observations and measurements. A total of 20 coated concrete specimens and 20 coated clay brick specimens were exposed.

Specimen observations were made for physical changes in the coating and at the holidays, as well as specimen weight changes. The results of the physical observations are summarized in Table 4-2, with photographs of typical specimens shown in Figures 4-1 and 4-2. Detailed observations for all of the specimens are included in Appendix B.

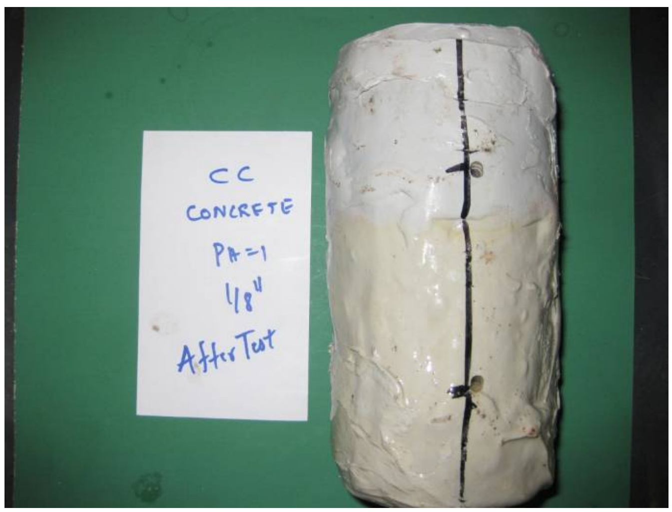


Figure 4-1. Concrete cylinder holiday specimen exposed to 1% H_2SO_4 solution.

Figure 4-2. Clay brick holiday specimen exposed to 1% H_2SO_4 solution.

Table 4-2. Summary of Chemical Exposure Observations (Protective Liner Systems PLS-614)

Specimen Material (Coating Condition)	DI Water				1% H_2SO_4 Solution				Comments
	Without Holidays		With Holidays		Without Holidays		With Holidays		
	30 days	180 days	30 days	180 days	30 days	180 days	30 days	180 days	
Concrete (Dry)	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Coating color change noted in portion submerged in acid solution.
Concrete (Wet)	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Coating color change noted in portion submerged in acid solution.
Clay Brick (Dry)	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Coating color change noted in portion submerged in acid solution.
Clay Brick (Wet)	N (2)	N (2)	N (2)	N (2)	N (2)	N (2)	N (4)	N (4)	Coating color change noted in portion submerged in acid solution.

N = No blister or crack.

(n) = Number of observed specimens.

As noted in the observations in Appendix B, there was discoloration of the coating noted in the portion of the specimens submerged in the acid solution, with less discoloration in the portion of the specimens exposed to acid vapor. There was no discoloration noted for the water exposed specimens. Likewise, there were no observed changes in the dimensions of any of the specimens at the holiday level. Weight changes were also monitored for the specimens, as summarized in Table 4-2.

Table 4-3. Average Specimen Weight Gain (%) After 180 Days of Immersion

Specimen Type	Holiday	Dry Coated (% weight gain)		Wet Coated (% weight gain)	
		DI Water	H_2SO_4	DI Water	H_2SO_4
Concrete	None	0.19	0.52	0.11	0.40
	⅛ in.	0.36	0.95	0.46	0.65
	½ in.	-	1.2	-	0.48
Clay Brick	None	0.58	6.6	0.66	2.2
	⅛ in.	6.6	6.7	5.0	6.4
	½ in.	-	6.6	-	5.1

4.1.2.2 Bonding Strength

Bonding strengths of the Protective Liner Systems CCP coating (dry and wet) with wet concrete and clay brick were determined according to CIGMAT CT-2 and CIGMAT CT-3 testing methods. All the coated specimens were cured under water. Both dry and wet concrete and clay brick specimens were coated to simulate the various field conditions. Performance of Coating PLS-614 was evaluated starting with application of the coating on March 9, 2009. The first bonding tests were completed approximately three weeks after application, around March 28, 2009. The other tests completed around June 28, 2009 (3 month samples) and September 28, 2009 (6 month samples). A total of 24 bonding tests with concrete specimens and 24 with clay brick specimens were completed.

Two of the failure modes (Type-1 and Type-4) involve substrate failure, whether entirely or in association with a bonding failure, while the other three failure modes are associated with either bonding or coating failures, whether singly or in combination. The actual coating bonding strength for failures involving substrate would be greater than indicated by the bonding strengths reported for Type-1 failures, as the bond of the coating exceeded the strength of the substrate (concrete or clay brick). Type-4 failures, which also involve substrate failure, are not as easily defined, as failure of the substrate could cause the coating to lose bond, or the loss of coating bond could result in a substrate failure.

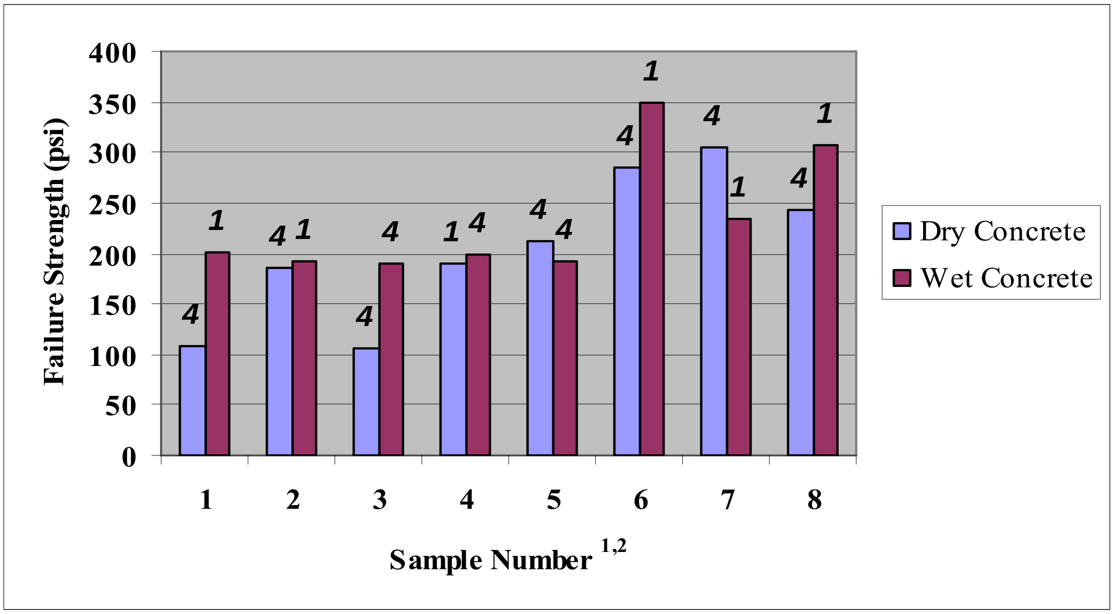
The results for all bonding strength tests, both concrete and clay brick, are summarized in Table 4-4. Further detail of bonding strengths for concrete specimens, wet and dry, are presented in

Figures 4-3 and 4-4, respectively. Bonding strength details for dry and wet clay bricks are presented in Figures 4-5 and 4-6, respectively. Photographs of typical failures are shown in Figures 4-7 through 4-9. Detailed descriptions of the results are summarized in Appendix C.

Table 4-4. Summary of Test Results for Bonding Strength Tests

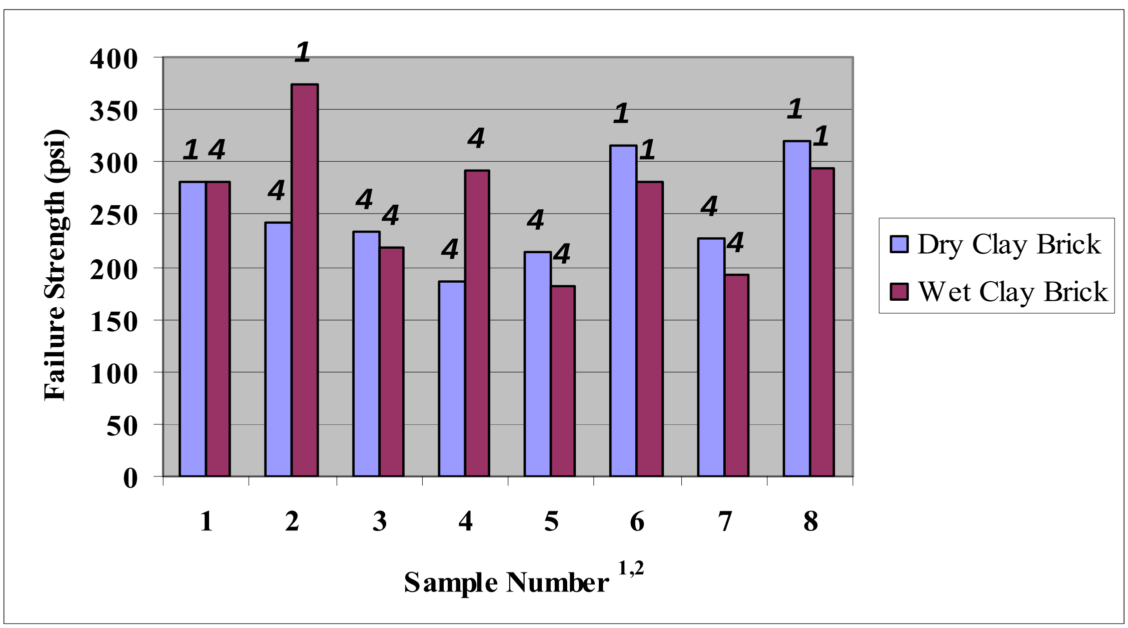
Substrate – Application Condition	Test [1]	Failure Type [2] – Number of Failures					Failure Strength (psi)	
		1	2	3	4	5	Range	Average
Concrete – Dry	Sandwich	4					232 – 293	269
	Pull-off	1			7		107 – 304	205
Concrete – Wet	Sandwich	3			1		257 – 321	287
	Pull-off	5			3		190 – 350	234
Clay Brick – Dry	Sandwich	4					314 – 350	335
	Pull-off	3			5		187 – 321	253
Clay Brick – Wet	Sandwich	4					338 – 384	366
	Pull-off	3			5		181 – 374	264

[1] Sandwich test (CIGMAT CT-3) or Pull-off test (CIGMAT CT-2).
[2] See Table 3-5.



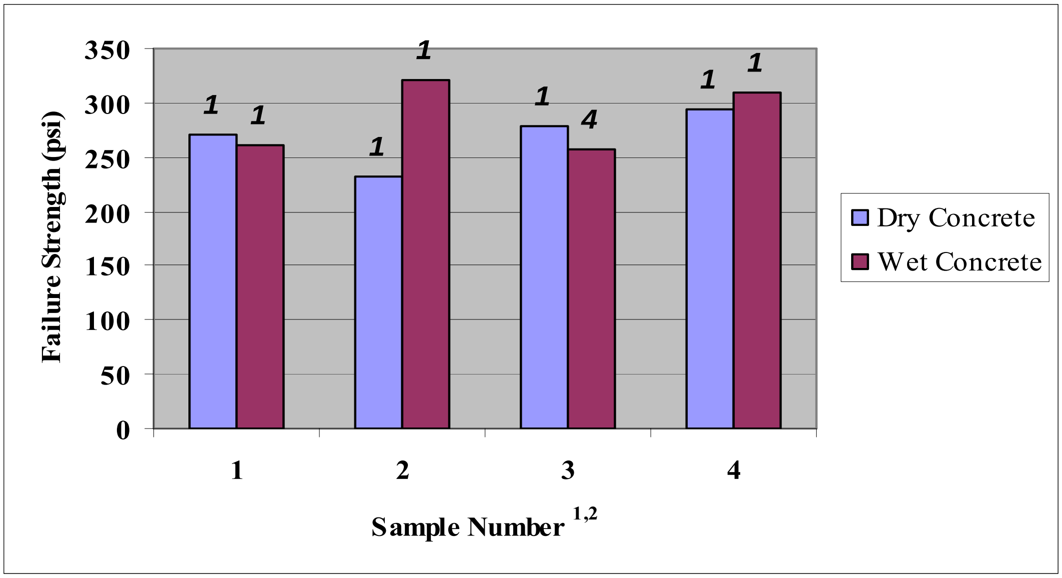
[1] Sample numbers 1 through 4 are 30-day breaks
Sample numbers 5 and 6 are 90-day breaks
Samples 7 and 8 are 180-day breaks
[2] ***Bold number*** above each column indicates Failure Type

Figure 4-3. Concrete bonding strength – pull-off test.



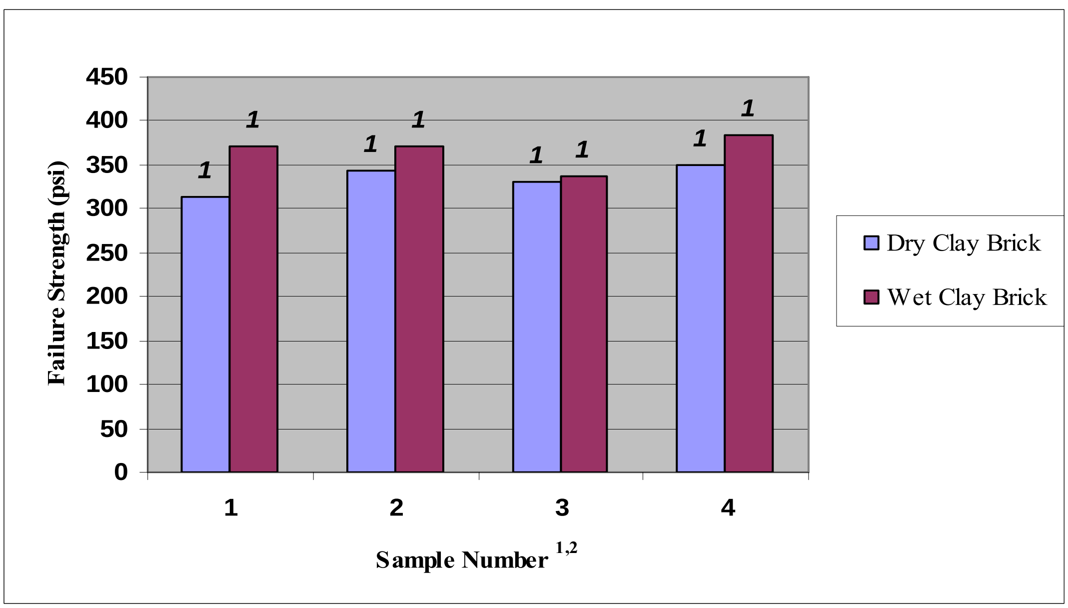
[1] Sample numbers 1 through 4 are 30-day breaks
Sample numbers 5 and 6 are 90-day breaks
Sample numbers 7 and 8 are 180-day breaks
[2] ***Bold number*** above each column indicates Failure Type

Figure 4-4. Clay brick bonding strength – pull-off test.



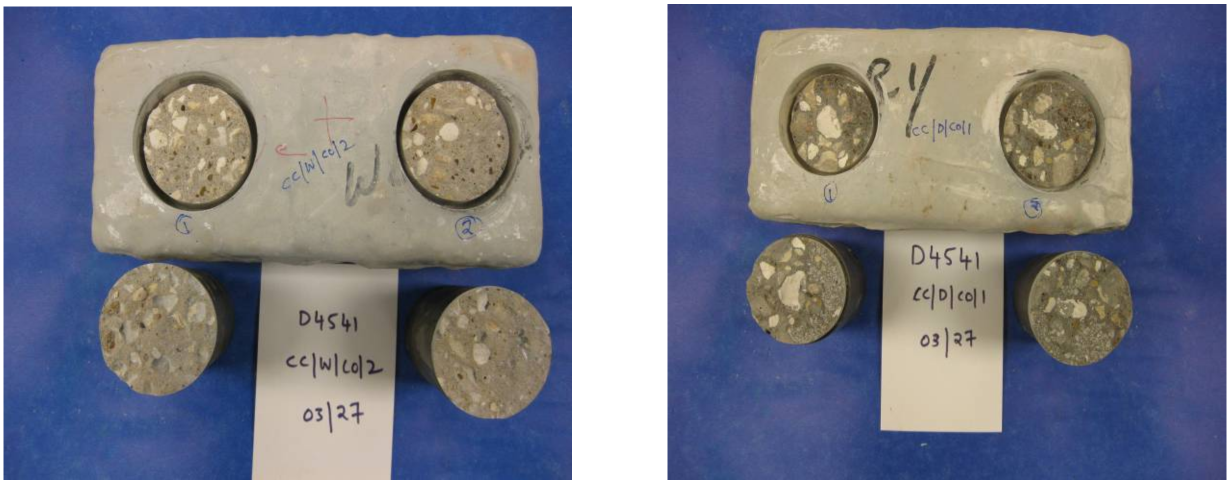
[1] Sample numbers 1 and 2 are 30-day breaks
Sample number 3 is the 90-day break
Sample number 4 is the 180-day break
[2] ***Bold number*** above each column indicates Failure Type

Figure 4-5. Concrete bonding strength – sandwich test.



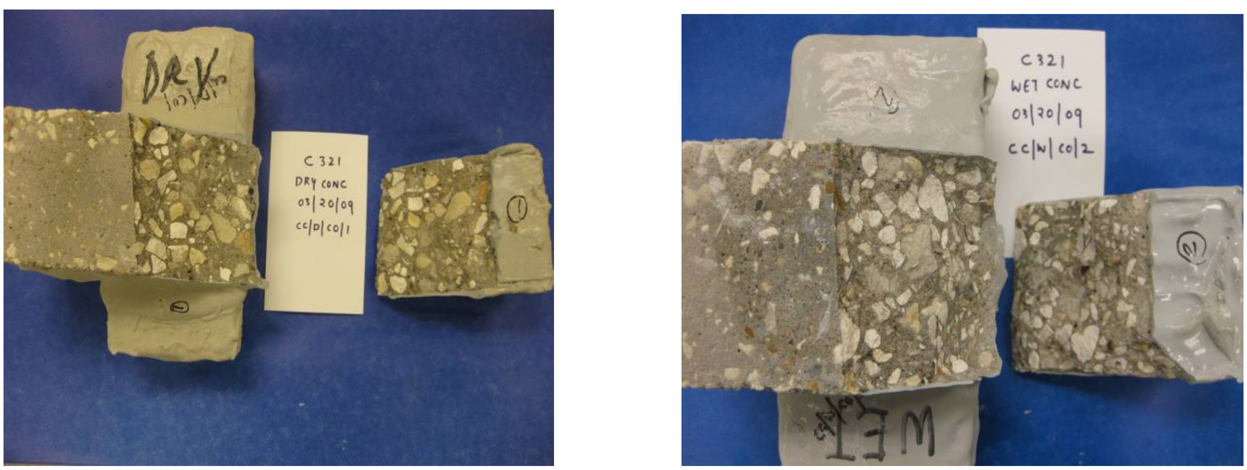
[1] Sample numbers 1 and 2 are 30-day breaks
Sample number 3 is the 90-day break
Sample number 4 is the 180-day break
[2] ***Bold number*** above each column indicates Failure Type

Figure 4-6. Clay brick bonding strength – sandwich test.



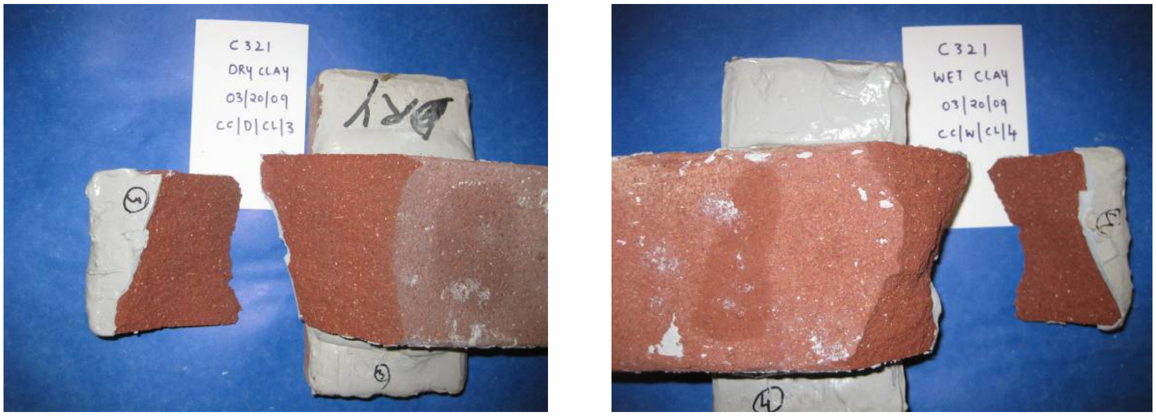
(a) Wet concrete (b) Dry concrete

Figure 4-7. Type-3 and Type-1 failure during CIGMAT CT-2 test with (a) wet and (b) dry concrete, respectively.



(a) Dry PLS-614 coated concrete (b) Wet PLS-614 coated concrete

Figure 4-8. Type-1(a) and Type-5 (b) failures during CIGMAT CT-3 test – (a) dry-coated concrete and (b) wet-coated concrete.



(a) Dry PLS-614 coated clay brick **(b) Wet PLS-614 coated clay brick**

Figure 4-9. Bonding failure (Type-1 failure) during CIGMAT CT-3 test – (a) dry-coated clay brick and (b) wet-coated clay brick.

4.2 Summary of Observations

A combination of laboratory tests was used to evaluate the performance, over a six-month period, of Protective Liner Systems, Inc. Epoxy Mastic PLS-614 (dry and wet) for coating concrete and clay bricks. The following observations are based on the testing results:

<u>**General Observations**</u>

- Samples of the coating product showed no weight gain when exposed to water over a 10-day period.
- None of the coated concrete or clay brick specimens, with or without holidays, showed any indication of blisters or cracking during the six-month holiday-chemical resistance tests.
- There were no observed changes in the dimensions of the coated concrete or clay brick specimens at the holiday levels for either DI or acid exposures.
- All 48 of the bonding tests resulted in substrate and substrate/bonding failures, with 27 substrate failures (Type-1) and 21 bonding/substrate failures (Type-4).

<u>**Concrete Substrate**</u>

- Weight gain was < 0.60% for any of the coated concrete specimens without holidays.
- Weight gain was <1.0% for any of the coated specimens with holidays for both water and acid exposures.
- Dry-coated concrete failures were mostly (7 of 12) bonding and concrete substrate (Type -4) failures, with the remainder being concrete substrate (Type-1) failures.
- Average tensile bonding strength for dry-coated concrete specimens was 226 psi, with individual specimens ranging from 107 to 304 psi.

- Wet-coated concrete failures were mostly (8 of 12) concrete substrate (Type-1) failures, with the remainder being bonding and concrete substrate (Type-4) failures.
- Average tensile bonding strength for wet-coated concrete specimens was 252 psi, with individual specimens ranging from 190 to 350 psi.

Clay Brick Substrate

- Without holidays, weight gain was < 0.45% for water exposed coated clay brick specimens; weight gain for acid exposed coated clay brick specimens was about 2.2-6.6%.
- With holidays, weight gains were > 5% for water exposed specimens and generally > 6% for acid exposed specimens; the holiday size did not make a significant difference in weight gain.
- Dry-coated clay brick failures were mostly (7 of 12) clay brick substrate (Type -1) failures, with the remaining five being bonding and clay brick substrate (Type-4) failures.
- Average tensile bonding strength for dry-coated clay brick specimens was 280 psi, with individual specimens ranging from 187 to 350 psi.
- Wet-coated clay brick failures were predominantly (7 of 12) clay brick substrate (Type-1) failures, with the remaining five being bonding and clay brick substrate (Type-4) failures.
- Average tensile bonding strength with wet-coated clay brick was 286 psi, with individual specimens ranging from 181 to 384 psi.

SECTION 5
QA/QC RESULTS AND SUMMARY

The VTP included a Quality Assurance Project Plan (QAPP) that identified critical measurements for this verification. The verification test procedures and data collection followed the QAPP to ensure quality and integrity. The Center for Innovative Grouting Materials and Technology (CIGMAT) was primarily responsible for implementing the requirements of the QAPP during testing, with oversight from NSF.

The QAPP identified requirements for preparation of the concrete and clay brick specimens that would be coated and used during the verification, along with requirements for quality control indicators (representativeness, completeness and precision) and auditing.

5.1 Specimen Preparation

For each batch of concrete made at CIGMAT and clay bricks purchased to perform the laboratory tests, specimens were tested to be sure their properties were within allowable ranges. The tests included unit weight, pulse velocity and water absorption of the specimens. Flexural and compressive strengths were also measured, where appropriate, to characterize the specimens. The target values for the specimens were maximum or minimum value of the batch within ±20% of the mean value of the batch. The property ranges for the different materials are summarized in Table 5-1.

Table 5-1. Typical Properties for Concrete and Clay Brick Specimens

Material	Unit Weight (pcf)	Pulse Velocity (fps)	Strength (psi)		Water Absorption (%)
			Compressive	Flexural	
Concrete	117-172	12,700-15,800	4000-5000	900-1300	0.5-2
Clay Brick	132-153	8,500-10,250	NA	700-1200	18-30

5.1.1 Unit Weight and Pulse Velocity

5.1.1.1 Concrete

The pulse velocity and unit weight were determined for 20 concrete cylinders and 36 concrete prisms. The unit weight of the concrete cylinder specimens varied between 127 pcf (2034 kg/m^3) and 150 pcf (2403 kg/m^3), with a mean value of 144 pcf (2307 kg/m^3). The allowable range (±20% of the mean value of the batch) is 102 pcf to 180 pcf. The concrete cylinder specimens fell within this range. Pulse velocities ranged from 12,700 fps to 15,800 fps, with a mean of 13,600 fps, within the allowable range of 20% of the mean value of the batch.

For the concrete block specimens, the unit weight varied between 117 pcf (1874 kg/m^3) and 172 pcf (2755 kg/m^3), with a mean value of 141 pcf (2259 kg/m^3). The allowable range ($\pm$20% of the mean value of the batch) is 94 pcf to 206 pcf. The concrete block specimens fell within this range. Pulse velocities ranged from 13,100 fps to 15,200 fps, with a mean of 13,700 fps, within the allowable range of 20% of the mean value of the batch.

There was no direct correlation between the pulse velocity and unit weight of concrete (Figure A1(a)). The variation of pulse velocity was normally distributed (Figure A1(b)).

5.1.1.2 Clay Brick

The unit weight and pulse velocity were determined on 56 clay brick specimens. The unit weight of clay brick specimens varied between 132 pcf (2114 kg/m^3) and 153 pcf (2451 kg/m^3), with a mean value of 138 pcf (2211 kg/m^3) . The specimens all fell within the $\pm$20% of the mean value of the batch.

The pulse velocity varied from 8,500 fps to 10,250 fps. There was no direct correlation between the pulse velocity and unit weight of clay bricks (Figure A2(a). The variation of pulse velocity was normally distributed (Figure A2(b)).

5.1.2 Water Absorption

5.1.2.1 Concrete

The chemical resistance (DI water and an H_2SO_4 solution) of the concrete specimens was determined using one dry and one wet cylinder. The cylinders were partially submerged (50%) in the liquid solutions and each was weighed after 10, 30 and 60 days. The dry concrete cylinder partially submerged (50%) in water showed continuous increase in weight up to 0.4% in 60 days, while the wet concrete in water showed a 0.1% increase in weight in 60 days. Initially (30 days), the specimens showed a slight weight gain in the H_2SO_4 solution, but over 60 days a weight loss, with visible corrosion, was observed in both the dry and wet concrete specimens. The overall weight loss was about 0.5%. Results are summarized in Appendix A, Tables A1 and A2 for concrete cylinders dry and wet, respectively.

5.1.2.2 Clay Bricks

Dry bricks in both water and acid solutions showed similar weight gain of 13% and 15%, respectively, over the 60 days of exposure. Wet bricks showed much smaller weight gain compared with the dry bricks, with 0.4% and 0.5% gains for the water and acid exposures, respectively. Weight increase was not observed with further soaking. Results are summarized in Appendix A, Tables A3 and A4 for dry and wet clay brick, respectively.

5.1.3 Compressive and Flexural Strength

While not required by the VTP, compressive and flexural strengths were determined for the concrete and clay brick specimens, as appropriate. This information provides further assurance that the specimens are acceptable for this verification.

5.1.3.1 Concrete

Two specimens each of dry and wet concrete cylinders were tested for compressive strength, and two wet and two dry concrete block specimens were tested for flexural strength. All specimens were cured for 28 days. The average compressive strength was about 5900 psi (41 MPa) for the wet concrete and about 4100 psi (28 MPa) for the dry cured concrete. The average flexural strength for the wet concrete was about 1200 psi (8.3 MPa) and about 1100 psi (7.6 MPa) for the dry concrete. Compressive and flexural strengths of dry and wet concrete are summarized in Table A5 in Appendix A.

5.1.3.2 Clay Brick

The average flexural strength was about 1100 psi (7.6 MPa) and about 930 psi (6.4 MPa) for wet dry and wet clay bricks, respectively. The flexural strength is important for bonding test CIGMAT CT-3 (Modified ASTM C321-94). The flexural strengths of the dry and wet clay bricks are summarized in Appendix A, Table A5.

5.2 Quality Control Indicators

5.2.1 Representativeness

Representativeness of the samples during this evaluation was addressed by CIGMAT personnel following consistent procedures in preparing specimens, having the vendor apply coatings to the specimens, and following CIGMAT SOPs in curing and testing of the coated specimens.

5.2.2 Completeness

The numbers of substrate and coating specimens to be evaluated during preparation of the test specimens, as well as the number of coated specimens to be tested during the verification, were described in the VTP. The numbers that were completed during the verification testing are described in this section.

5.2.2.1 Specimen Preparation

The number (per the VTP) of each specimen to be used for characterization of the substrates is listed in Table 5-2. As there were multiple coatings being evaluated at the same time, CIGMAT prepared a batch of specimens to be coated in the tests. The number of specimens characterized during preparation of the batch of specimens is indicated in parentheses for each material and test listed in Table 5-2.

Table 5-2. Number of Specimens Used for Each Characterization Test

Material	Number of Specimens Used in Test				
	Unit weight	**Pulse velocity**	**Water absorption**	**Flexure***	**Compression ***
Coating	6 (6)	6 (6)	6 (6)	None	None
Concrete Cylinders	20 (102)	20 (18)	10 (10)	None	3
Concrete Prisms	36 (189)	36 (37)	None	3	None
Clay Prisms (Brick)	56 (159)	56 (18)	10 (10)	3	None

* Flexure and compression tests were performed for informational purposes only.

The number of specimens tested meet, or exceed the VTP requirement except for the pulse velocity for concrete cylinders and clay bricks. The unit weight of concrete is the most important parameter to determine the quality of the concrete, so every sample was tested for unit weight. The pulse velocity test, a special test not available for routine testing in test laboratories, was used at CIGMAT to randomly check the quality of the concrete. The pulse velocity test results on randomly selected concrete samples showed that there was nothing unusual about the concrete samples that were tested. As summarized in Appendix A, there was no direct correlation between the pulse velocity and unit weight of concrete, and the variation of pulse velocity was normally distributed.

The clay bricks obtained for testing were from the same batch. Quality control for the clay bricks involved both unit weight measurements and pulse velocity testing. The unit weight of each brick was determined, while the pulse velocity testing was completed on a random selection of bricks from the entire batch. The unit weights showed that there was nothing unusual (voids) in the specimens. The pulse velocity test was completed on 18 bricks (not the 56 indicated in the VTP). CIGMAT, based on their experience in testing with clay bricks, determined that the results of the 18 tests, combined with the unit weight data, were adequate to characterize the quality of the bricks. As summarized in Appendix A, there was no direct correlation between the pulse velocity and unit weight of clay bricks, and the variation of pulse velocity was normally distributed.

5.2.2.2 Coating Testing

The number (per the VTP) of coated specimens evaluated for each substrate during the testing is indicated in Table 5-3. The number of coated specimens was the same for each material (concrete or clay brick) and is indicated in parentheses in Table 5-3. The bonding tests were completed over a period of six months to determine if there were changes in bonding strength with time. Normally, the 3- and 6-month bonding test results do not differ much in failure type or bonding strength from the first tests (completed in the first 30 days), so additional specimens were evaluated at the initial test and fewer at later test times. The total number of specimens for the entire test was the same as indicated in the VTP.

Table 5-3. Total Number of Tests for Each Substrate Material

Exposure Time	Holiday Test (1)		Bonding Strength Test (2)	
	DI Water	1% H_2SO_4	Sandwich	Pull-Off
2 Weeks (3)			4 (4)	4 (8)
1 Month	8 (10)	12 (10)		
3 Months			4 (2)	4 (4)
6 Months	8 (10)	12 (10)	4 (2)	4 (4)

(1) The same specimens are monitored for 6 months.
(2) The number of dry-or wet-coated specimens is the same, and equal to half of the number indicated.
(3) The bonding tests were completed at 30 days during testing.
(n) = Number of specimens observed or tested.

5.2.3 Precision

As specified in Standard Methods (Method 1030 C), precision is specified by the standard deviation of the results of replicate analyses. The overall precision of a study includes the random errors involved in sampling as well as the errors in sample preparation and analysis. The VTP did not establish objectives for this measure.

In this evaluation, analysis is made using two different substrate materials (concrete and clay brick), each under two different conditions (dry-coated and wet-coated). Comparison of the results for multiple specimens prepared under similar conditions provides some indication of the variability of the bonding tests. For most of the bonding tests, there were only one or two specimens prepared and cured in the same manner and duration. The results for the 30-day pull-off tests, where there were four samples analyzed for each substrate and condition, are compared. The results are shown in Table 5-4.

Table 5-4. Standard Deviation for 30-Day Pull-Off Test

Substrate – Condition	Number of Samples	Average Failure Strength (psi)	Standard Deviation (psi)
Concrete – Dry	4	148	46.5
Concrete – Wet	4	196	5.7
Clay brick – Dry	4	236	38.6
Clay brick – Wet	4	291	64.1

5.3 Audit Reports

NSF conducted an audit of the CIGMAT Laboratory prior to the verification test. The laboratory audit found that CIGMAT had the necessary equipment, procedures, and facilities to perform the coatings verification test described in the VTP. Systems were in place to record laboratory data and supporting quality assurance data obtained during the tests. Specialized log sheets were prepared for each of the procedures and these data sheets were stored with the study Director, Dr. Vipulanandan. This is important as some of these tests were performed over several months with extended periods between testing. The primary weakness identified in the CIGMAT systems was in documentation of the calibration and maintenance of the basic equipment. It was quite clear that calibration of the balances, pH meters, pulse velocity meter, etc. was indeed performed. All of the needed calibration reference standards and standard material s were available near each piece of equipment. However, the frequency of calibration and the actual calibration could not be verified as in most cases the information was not being recorded either on the bench sheet or in an equipment calibration notebook. A corrective action recommendation was made by NSF following the audit. A second site visit for a data review meeting after the testing was completed indicated that CIGMAT instituted a system for recording calibrations during the testing period.

SECTION 6
REFERENCES

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APPENDIX A

Data from Evaluation of Pre-Coated Test Specimens

Behavior of Concrete, Clay Brick and Coating
Summary

In order to ensure the quality, the concrete (cylinders and blocks) and clay bricks used in this study were tested; the results are summarized in this section. Also, samples of the coating product itself were analyzed to characterize the coatings.

A. 1. ***Unit Weight and Pulse Velocity***

To ensure the quality of the concrete and clay brick specimens used in this coating study the unit weight and pulse velocity of the specimens were measured. Six specimens of the coating were evaluated for unit weight, pulse velocity and water absorption to provide basic data to verify that the coating used in any future application is the same as applied for this verification testing.

Concrete: The variation of pulse velocity with unit weight is shown in Figure A1. The unit weight of concrete specimens varied between 117 pcf (1874 kg/m^3) and 172 pcf (2756 kg/m^3). The pulse velocity varied from 12,700 fps to 15,800 fps. There was no direct correlation between the pulse velocity and unit weight of concrete (Figure A1(a)). The variation of pulse velocity was normally distributed (Figure A1(b)).

Clay Brick: The variation of pulse velocity with unit weight is shown in Figure A2. The unit weight of clay brick specimens varied between 132 pcf (2115 kg/m^3) and 153 pcf (2451 kg/m^3). The pulse velocity varied from 8500 fps to 10,250 fps. There was no direct correlation between the pulse velocity and unit weight of clay bricks (Figure A2(a). The variation of pulse velocity was normally distributed (Figure A2(b)).

Coating: The unit weight of coating varied from 63 pcf to 68 pcf with an average of 65 pcf with a coefficient of variation of 1.9%. The pulse velocity varied from 8660 fps to 8990 fps with an average of 8791 fps with a coefficient of variation of 1.3% (Table A6).

A. 2. ***Chemical Resistance***

Concrete: Chemical resistant results are summarized in Tables A1 and A2 for concrete cylinders dry and wet respectively. Dry concrete cylinders partially submerged (50%) in water showed continuous increase in weight up to 0.4% in sixty days. The wet concrete in water showed a 0.1% increase in weight in 60 days. Weight loss and visible corrosion were observed in the dry and wet concrete specimens in the sulfuric acid solution (pH = 1).

Clay Bricks: Results are summarized in Tables A3 and A4 for dry and wet clay brick, respectively. Dry bricks in water and acid showed similar gains in weight of over 10%. No visible damage to the bricks was observed. Wet bricks showed much smaller weight gain as compared to the dry bricks. Weight increase was not observed with further soaking.

Coating: Specimens immersed in water for 10 days showed no gain in weight.

A. 3. ***Strength***

Concrete: Results for the compressive and flexural strength of dry and wet concrete are summarized in Table A5 in Appendix A. The minimum compressive strength of 28 days water-cured concrete was 4100 psi (28 MPa) and the flexural strength was 1065 psi (7.6 MPa).

Clay Brick: Flexural strength of dry and wet clay bricks are summarized in Table A5 in Appendix A. The average flexural strength was 1136 psi and 932 psi for wet dry and wet clay bricks. The flexural strength is important for bonding test CIGMAT CT-3 (Modified ASTM C321-94).

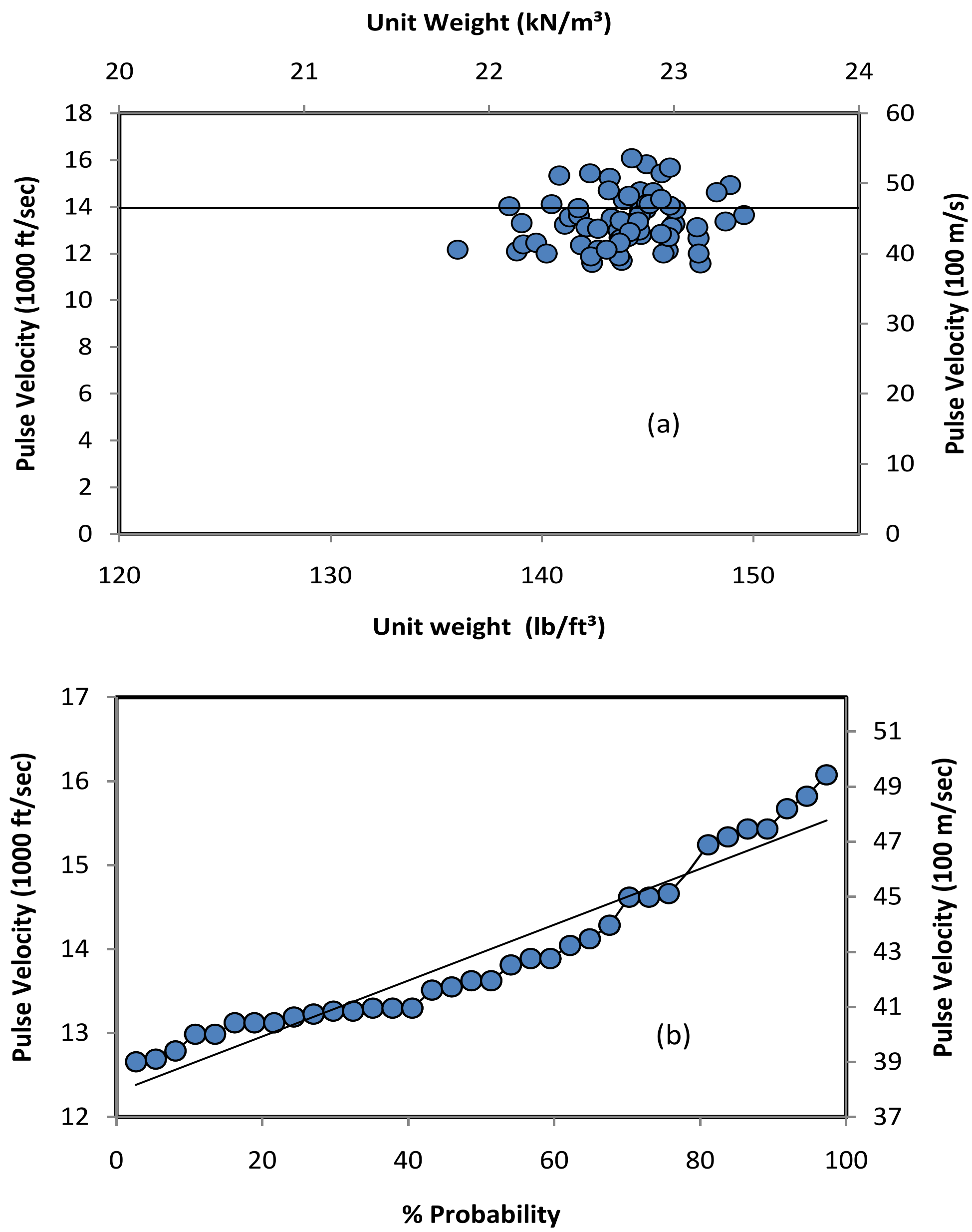


Figure A1. Quality control for concrete brick specimens (a) pulse velocity versus unit weight and (b) distribution of pulse velocity.

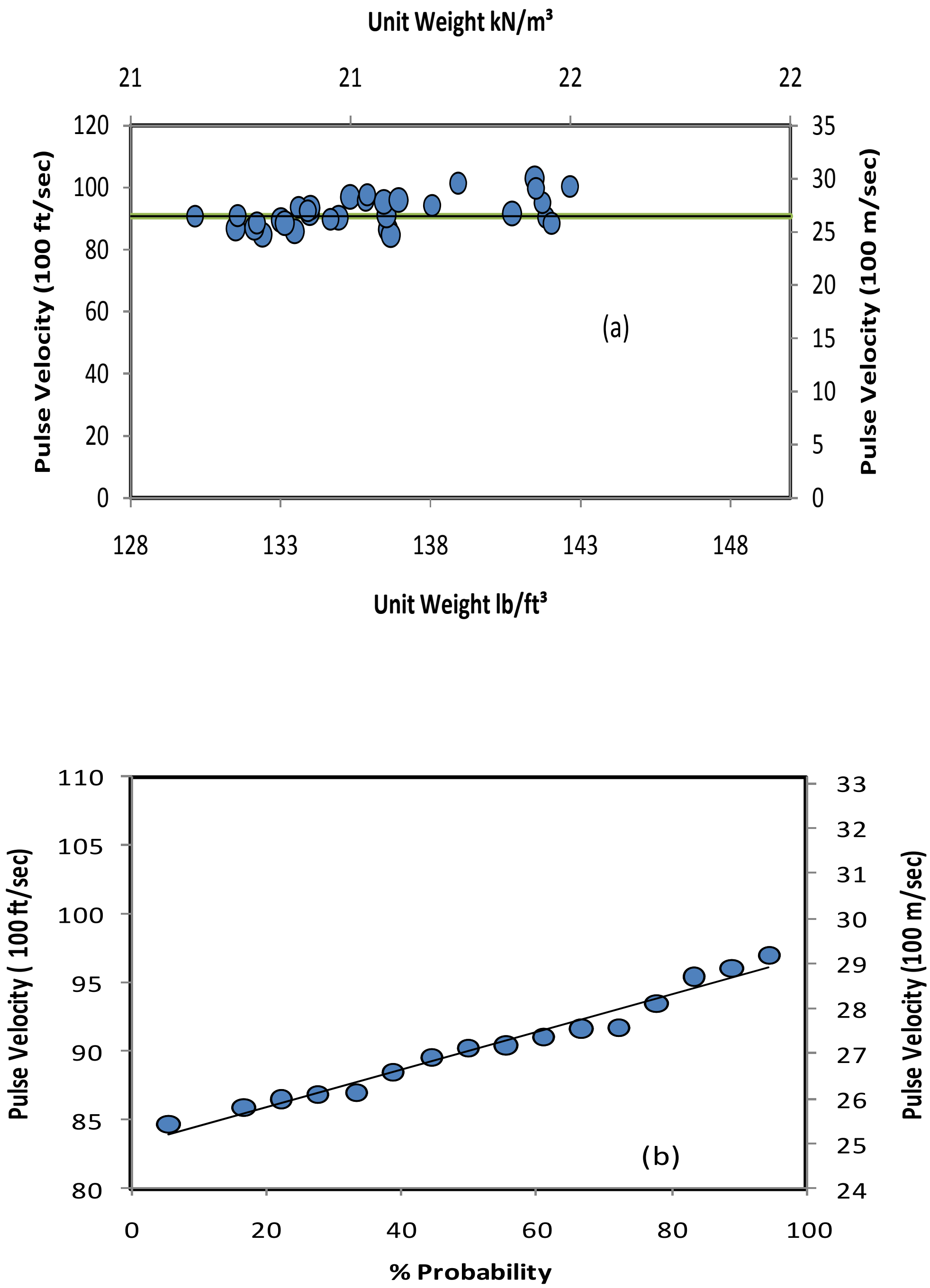


Figure A2. Quality control for clay brick specimens (a) pulse velocity versus unit weight and (b) distribution of pulse velocity.

Table A1. Results from Chemical Attack Test* on Dry Concrete (CIGMAT CT-1: No Holiday)

Concrete	Immersion Time (days)	Weight Change (%)		Remarks
		DI Water (pH= 6)	H_2SO_4 Solution (pH = 1)	
Dry	10	0.14	0.12	Similar weight change
	30	0.27	0.32	Similar weight change
	60	0.38	-0.48	Weight loss in acid solution
Remarks	Tested up to 2 months	Total weight change is 0.38 %	Total weight change is - 0.48%	Weight loss in H_2SO_4 solution in 60 days indicates the corrosivity

*50 % of specimen was submerged in liquid.

Table A2. Results from Chemical Attack Test* on Wet Concrete (CIGMAT CT-1: No Holiday)

Concrete	Immersion Time (days)	Weight Change (%)		Remarks
		DI Water (pH= 6)	H_2SO_4 Solution (pH = 1)	
Wet	10	0.06	0.11	Less weight gain in water
	30	0.09	0.31	Less weight gain in water
	60	0.11	-0.52	Weight loss in acid solution
Remarks	Tested up to 2 months	Total weight change is 0.11 %	Total weight change is -0.52 %	Weight loss in H_2SO_4 solution in 60 days indicates the corrosivity

*50 % of specimen was submerged in liquid.

Table A3. Results from Chemical Attack Test* on Dry Clay (CIGMAT CT-1: No Holiday)

Clay Brick	Immersion Time (days)	Weight Change (%)		Remarks
		DI Water (pH= 6)	H_2SO_4 Solution (pH = 1)	
Dry	10	9.9	9.0	Similar weight change
	30	13.6	15.6	Similar weight change
	60	14.9	17.6	Similar weight change
Remarks		Total weight change is 15 %	Total weight change is 18 %	Similar weight change in water and acid solution

*50 % of specimen was submerged in liquid.

Table A4. Results from Chemical Attack Test* on Wet Clay (CIGMAT CT-1: No Holiday)

Clay Brick	Immersion Time (days)	Weight Change (%)		Remarks
		DI Water (pH= 6)	H_2SO_4 Solution (pH = 1)	
Wet	10	0.18	0.25	Similar weight change
	30	0.32	0.43	Similar weight change
	60	0.40	0.52	Similar weight change
Remarks		Total weight change is 0.4 %	Total weight change is 0.52 %	Similar weight change in water and acid solution

*50 % of specimen was submerged in liquid.

Table A5. Minimum and Maximum Strengths of Concrete Cylinders, Blocks and Clay Bricks

Materials	Curing Time (days)	Compressive Strength (psi)		Flexural Strength (psi)	
		Wet	Dry	Wet	Dry
Concrete Cylinder (No. Specimens)	28	5893 (2)	4099 (2)	N/A	N/A
Concrete Block (No. Specimens)	28	N/A	N/A	1065 (2)	1167 (2)
Clay Brick (No. Specimens)	N/A	N/A	N/A	1136 (2)	932 (2)
Remarks	Concrete cured for 28 days	Information for quality control	Information for quality control	Related to ASTM C321-94 bonding test	Related to ASTM C321-94 bonding test

APPENDIX B

Test Results and Observations from Chemical Exposure – Holiday Test

Laboratory Test: Holiday Test
(CIGMAT CT-1 (Modified ASTM G 20-88))

Summary: Sulfuric Acid Resistance

In order to evaluate the performance of PLS-614, coated concrete cylinders and clay bricks were tested with and without holidays in water and sulfuric acid solution (pH=1). Performance of PLS-614 was evaluated over a period of six months from March 2009 to September 2009 in this study. A total of 20 coated concrete specimens and 20 coated clay brick specimens were tested. The results are summarized in Tables B1 through B6.

PLS-614 (Dry Coated)

(i) Concrete

One month (30 days): None of the specimens showed blisters or cracking. Mild change in color of the coating was observed in the portion of the specimens submerged in sulfuric acid solution (Table B.1)

Six months (180 days): None of the specimens showed blisters or cracking. Discoloration (noteable change) was observed in the lower part of the specimens (liquid phase) and partially in the upper part of the specimens (vapor phase), immersed in sulfuric acid solution (Table B.3).

(ii) Clay Brick

One month (30 days): None of the specimens showed blisters or cracking. Mild change in color of the coating was observed in the portion of the specimens submerged in sulfuric acid solutions.

Six months (180 days): None of the specimens showed blisters or cracking. Discoloration was observed on the portion of the specimens submerged in sulfuric acid solutions.

PLS-614 (Wet Coated)

(i) Concrete

One month (30 days): None of the specimens showed blisters or cracking. Minor change in color of the coating was observed in the portion of the specimens submerged in sulfuric acid (Table B.2)

Six months (180 days): None of the specimens showed blisters or cracking. Discoloration (DC) was observed in the lower part of the specimens (liquid phase) and partially in the upper part of the specimens (vapor phase) immersed in sulfuric acid solution (Table B.4).

(ii) Clay Brick

One month (30 days): None of the specimens showed blisters or cracking. Minor change in color of the coating was observed on the portion of the specimens submerged in sulfuric acid solutions.

Six months (180 days): None of the specimens showed blisters or cracking. Discoloration was observed on the portion of the specimens submerged in sulfuric acid solutions.

Rating Criteria for Holiday Test Results

No Blister or Cracking (N): No visible blister. No discoloration. No cracking.
Blister (B): Visible blister up to one inch in diameter. No discoloration. No cracking.
Cracks (C): Blister with diameter greater than one inch and/or cracking of coating at the holiday.

Table B.1. Holiday Test Results for Protective Liner Systems PLS-614 Dry-Coated Concrete after 30 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Dry	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4(100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/0)	Total of 10 specimens tested
Remarks	After 30 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B2. Holiday Test Results for Protective Liner Systems PLS-614 Wet-Coated Concrete after 30 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Wet	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/0)	Total of 10 specimens tested
Remarks	After 30 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B.3. Holiday Test Results for Protective Liner Systems PLS-614 Dry-Coated Concrete after 180 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Dry	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/0)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B.4. Holiday Test Results for Protective Liner Systems PLS-614 Wet-Coated Concrete after 180 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Wet	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/00)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C=Cracking

Table B5. Holiday Test Results for Protective Liner Systems PLS-614 Dry-Coated Clay Brick after 180 Days Immersion (CIGMAT CT-1)

Clay	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Dry	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/6)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B6. Holiday Test Results for Protective Liner Systems PLS-614 Wet-Coated Clay Brick after 180 Days Immersion (CIGMAT CT-1)

Clay	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Wet	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/0)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B7. Holiday Test Results for Protective Liner Systems PLS-614 Dry-Coated Clay Brick after 180 Days Immersion (CIGMAT CT-1)

Clay	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Dry	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/6)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B8. Holiday Test Results for Protective Liner Systems PLS-614 Wet-Coated Clay Brick after 180 Days Immersion (CIGMAT CT-1)

Clay	Holiday	Medium and Rating (No. of Specimens)		Total No. % (N/B/C)	Remarks
		DI Water	1% H_2SO_4		
Wet	No Holiday	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/8 in.	N (2)	N (2)	4 (100/0/0)	Coating color changed in the acid submerged portion
	1/2 in.	---	N (2)	2 (100/0/0)	Coating color changed in the acid submerged portion
Total No. % (N/B/C)		4 (100/0/0)	6 (100/0/0)	10 (100/0/0)	Total of 10 specimens tested
Remarks	After 180 days immersion	100% N	100% N		No visible blisters or cracking; only coating color change noted.

N = No blisters or crack
B = Blister
C = Cracking

Table B9. Holiday Test Results for PLS-614 Dry-Coated Concrete Brick – Weight Change after 180 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Average Weight Change (%)		Remarks
		DI Water	H_2SO_4	
Dry	No Holiday	0.19	0.52	Greater weight change in acid
	1/8 in.	3.6	0.95	Higher weight change in water
	1/2 in.	--	1.2	Higher weight change with increased holiday size
Remarks	After 180 days immersion	Specimens with holiday showed greater weight change	Specimens with holidays showed no great weight change	Holidays increased the weight change in water exposed specimens; less so in acid exposed specimens.

Table B10. Holiday Test Results for PLS-614 Wet-Coated Concrete Brick – Weight Change after 180 Days Immersion (CIGMAT CT-1)

Concrete	Holiday	Average Weight Change (%)		Remarks
		DI Water	H_2SO_4	
Wet	No Holiday	0.11	0.40	Similar weight change
	1/8 in.	0.46	0.65	Similar weight change
	1/2 in.	--	0.48	Similar weight change with increased holiday size
Remarks	After 180 days immersion	Similar weight change	Similar weight change	Similar weight changes

Table B11. Holiday Test Results for PLS-614 Dry-Coated Clay Brick – Weight Change after 180 Days Immersion (CIGMAT CT-1)

Clay Brick	Holiday	Average Weight Change (%)		Remarks
		DI Water	H_2SO_4	
Dry	No Holiday	0.58	6.6	Higher weight change in acid
	1/8 in.	5.8	6.7	Similar weight change
	1/2 in.	--	6.6	Similar weight change with increased holiday size
Remarks	After 180 days immersion	Specimens with holiday showed greater weight change	Specimens with holidays showed similar weight change	Greater weight change with holidays in water exposed specimens; holidays did not increase change in acid exposed specimens.

Table B12. Holiday Test Results for PLS-614 Wet-Coated Clay Brick – Weight Change after 180 Days Immersion (CIGMAT CT-1)

Clay Brick	Holiday	Average Weight Change (%)		Remarks
		DI Water	H_2SO_4	
Wet	No Holiday	0.66	2.2	Higher weight change in acid
	1/8 in.	5.0	6.4	Higher weight change in acid
	1/2 in.	--	5.1	Less weight change with increased holiday size in acid
Remarks	After 180 days immersion	Specimens with holiday showed greater weight change	Specimens with holidays showed greater weight change	Holidays increased the weight change but the size of holiday did not affect weight change in acid

APPENDIX C

Results and Observations from Bonding Tests

Laboratory Test: Bonding Test
(CIGMAT CT-2, Modified ASTM D4541-85 and
CIGMAT CT-3, Modified ASTM C321-94)

Summary: Tensile Bonding Strength

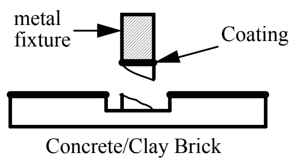
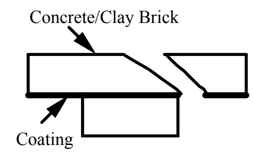
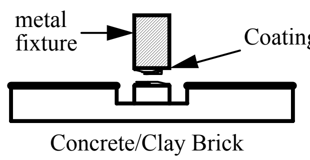
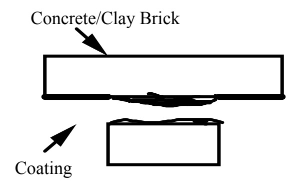
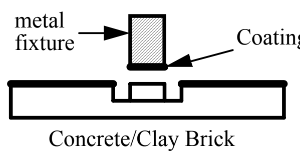
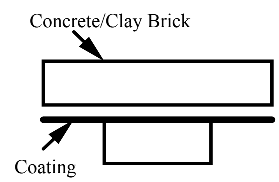
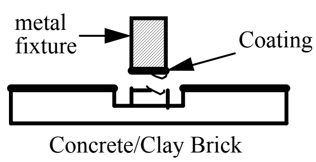
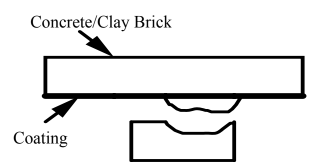
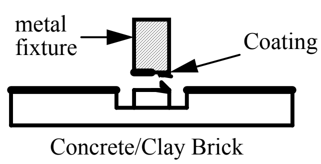
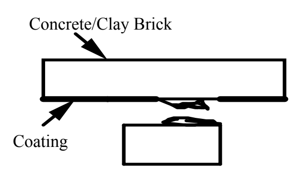
Total CIGMAT CT-2 Tests =24 Total CIGMAT CT-3 Tests = 16

Bonding strengths of coating PLS-614 (dry and wet) with concrete and clay brick were determined according to CIGMAT CT-2 (modified ASTM D4541-85) and CIGMAT CT-3 (modified ASTM C321-94) testing methods. All the coated specimens were cured under water. Both dry and wet specimens were coated to simulate the various field conditions. The performance of Coating PLS-614 was evaluated starting in February 2009; results are included in this report. A total of 24 bonding tests with concrete specimens and 24 with clay brick specimens were performed.

Failure Types

All the failure types encountered in the bonding tests (modified ASTM D 4541 and ASTM C 321) are listed in Table C1. Type-1 failure is substrate failure (Table C1). This is the most desirable result if the bonding strength is quite high (in the range 8% to 12% of the concrete substrate compressive strength). In Type-2 failure (Table C1), the coating has failed. Type-3 failure is bonding failure where failure occurred between the coating and substrate. Type-4 and Type-5 are combined failures. Type-4 failure is the bonding and substrate failure where the failure occurs in the substrate and on the interface of the coating and the substrate. This indicates that the adhesive strength is comparable with the tensile strength of the substrate. Type-5 failure (Table C1) is coating and bonding failure where the failure occurs due to low cohesive and adhesive strength of the coating.

Table C1. Failure Types of Modified ASTM D 4541 Test and ASTM C 321 Test

Failure Type	Description	CIGMAT CT-2 Test (Modified ASTM D 4541)	CIGMAT CT-3 Test (Modified ASTM C 321)
Type-1	Substrate Failure		
Type-2	Coating Failure		
Type-3	Bonding Failure		
Type-4	Bonding and Substrate Failure		
Type-5	Bonding and Coating Failure		

PLS-614 (Dry Coating)

(i) Concrete

CIGMAT CT-2 (modified ASTM D 4541-85): A total of eight tests was performed, with all but one of the failures being Type-4. The other was a Type-1 failure. The bonding strengths ranged from 107 to 304 psi for both failure types. Type-4 failures ranged from 107 to 304 psi, while the Type-1 failure was 191 psi. The average bonding strength from the pull-off tests was 205 psi (1.4 MPa) (Table C2).

CIGMAT CT-3 (modified ASTM C 321-94): A total of four tests was performed, with all failures being Type-1. The bonding strengths ranged from 232 to 293 psi. The average bonding strength from the sandwich tests was 269 psi (1.8 MPa) (Table C6).

Summary: The type of test influenced the mode of failure and the bonding strength. Mostly Type-4 failures (7 of 8, with the other being Type-1) were observed during the pull-off test (CIGMAT CT-2), while the sandwich test (CIGMAT CT-3) produced all Type-1 failures. The average bonding strength from CIGMAT CT-2 tests was 205 psi (1.4 MPa) and from CIGMAT CT-3 tests was 269 psi (1.8 MPa). Average tensile bonding strength with dry concrete was 226 psi (1.6 MPa), ranging from 107 to 304 psi, with 58% (7 of 12) being bonding/substrate (Type-4) failures and the rest being substrate (Type-1) failures.

(ii) Clay Brick

CIGMAT CT-2 (modified ASTM D 4541-85): A total of eight tests was performed, with five of the failures being Type-4 and the other three being Type-1 failures. The bonding strengths ranged from 187 to 321 psi for both failure types. Type-4 failures ranged from 187 to 226 psi, while the Type-1 failures ranged from 281 to 321 psi. The average bonding strength from the pull-off tests was 253 psi (1.7 MPa) (Table C5).

CIGMAT CT-3 (modified ASTM C 321-94): A total of four tests was performed, with all failures being Type-1. The bonding strengths ranged from 314 to 350 psi. The average bonding strength from the sandwich tests was 335 psi (2.3 MPa) (Table C8).

Summary: The type of test influenced the mode of failure and the bonding strength. Mostly Type-4 failures (5 of 8), with the other three being Type-1, were observed during the pull-off test (CIGMAT CT-2), while the sandwich test (CIGMAT CT-3) produced all Type-1 failures. The average bonding strength from CIGMAT CT-2 tests was 253 psi (1.7 MPa) and from CIGMAT CT-3 tests was 335 psi (2.3 MPa). Average tensile bonding strength with dry clay brick was 280 psi (1.9 MPa), ranging from 187 to 350 psi, with 58% being substrate (Type-1) failures and the rest being bonding/substrate (Type-4) failures.

PLS-614 (Wet Coating)

(i) Concrete

CIGMAT CT-2 (modified ASTM D 4541-85): A total of eight tests was performed, with five of the 8 being Type-1 and the other three being Type-4 failures. The bonding strengths ranged from 190 to 350 psi for both failure types. Type-1 failures ranged from 193 to 350 psi, while the Type-4 failures ranged from 109 to 200 psi. The average bonding strength from the pull-off tests was 234 psi (1.6 MPa) (Table C3).

CIGMAT CT-3 (modified ASTM C 321-94): A total of four tests was performed. Three of the four were Type-1 failures, with the other being a Type-4 failure. The bonding strengths ranged from 262 to 321 psi for the Typ3-1 failures and 257 psi for the Type-4 failure. The average bonding strength from the sandwich tests was 287 psi (2.0 MPa) (Table C7).

Summary: The type of test influenced the mode of failure and the bonding strength. Mostly Type-1 failures (5 of 8), the other three being Type-4, were observed during the pull-off test (CIGMAT CT-2), while the sandwich test (CIGMAT CT-3) produced three Type-1 and one Type-4 failures. The average bonding strength from CIGMAT CT-2 tests was 234 psi (1.6 MPa) and from CIGMAT CT-3 tests was 287 psi (2.0 MPa). Average tensile bonding strength with wet concrete was 252 psi (1.9 MPa), ranging from 190 to 350 psi, with 67% being substrate (Type-1) failures and the rest being bonding/substrate (Type-4) failures.

(ii) Clay Brick

CIGMAT CT-2 (modified ASTM D 4541-85): A total of eight tests was performed, with five of the failures being Type-4 and the other three being Type-1 failures. The bonding strengths ranged from 181 to 374 psi for both failure types. Type-4 failures ranged from 181 to 292 psi, while the Type-1 failures ranged from 281 to 374 psi. The average bonding strength from the pull-off tests was 264 psi (1.8 MPa) (Table C5).

CIGMAT CT-3 (modified ASTM C 321-94): A total of four tests was performed, with all failures being Type-1. The bonding strengths ranged from 338 to 384 psi. The average bonding strength from the sandwich tests was 366 psi (2.5 MPa) (Table C9).

Summary: The type of test influenced the mode of failure and the bonding strength. Mostly Type-4 failures (5 of 8), with the other three being Type-1, were observed during the pull-off test (CIGMAT CT-2), while the sandwich test (CIGMAT CT-3) produced all Type-1 failures. The average bonding strength from CIGMAT CT-2 tests was 264 psi (1.8 MPa) and from CIGMAT CT-3 tests was 366 psi (2.5 MPa). Average tensile bonding strength with wet clay brick was 298 psi (2.0 MPa), ranging from 181 to 384 psi, with 58% being substrate (Type-1) failures and the rest being bonding/substrate (Type-4) failures.

Table C2. Bonding Strength of PLS-614 with Dry Concrete CIGMAT CT-2 (Pull-Off)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type-4	Type-5	
Dry Concrete	30	×××			×		148
	90				××		248
	180				××		274
Total No. (% Failure)		3 (38%)	0 (0%)	0 (0%)	5 (62%)	0 (0%)	Total of 8 tests
Remarks	Up to 180 days	Good bonding strength	None	None	Good bonding strength	None	Types 1 and 4 failures; average bonding strength for all tests – 205 psi (1.4 MPa).

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C3. Bonding Strength of PLS-614 with Wet Concrete CIGMAT CT-2 (Pull-off)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type- 3	Type-4	Type-5	
Wet Concrete	30	××			××		196
	90	×			×		272
	180	××					272
Total No. (% Failure)		5 (62%)	0 (0%)	0 (0%)	3 (38%)	0 (0%)	Total of 8 tests
Remarks	Up to six (6) months	Good bonding strength	None	None	Good bonding strength	None	Types 1 and 4 failures; average bonding strength for all tests – 142 psi (1.0 MPa).

Type-1 = Substrate failure
Type- 2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C4. Bonding Strength of PLS-614 with Dry Clay Brick CIGMAT CT-2 (Pull-off)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type- 4	Type-5	
Dry Clay Brick	30	×			×××		236
	90	×			×		265
	180	×			×		274
Total No. (% Failure)		3 (38%)	0 (0%)	0 (0%)	5 (62%)	0 (0%)	Total of 8 tests
Remarks	Up to 180 days	Good bonding strength	None	None	Good bonding strength	None	Types-1 and-4 failures; average bonding strength for all tests – 253 psi (1.7 MPa)

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C5. Bonding Strength of PLS-614 with Wet Clay Brick CIGMAT CT-2 (Pull-off)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type-4	Type-5	
Wet Clay Brick	30	×××			×		291
	90	×			×		231
	180	×			×		243
Total No. (% Failure)		5 (68%)	0 (0%)	0 (0%)	3 (38%)	0 (0%)	Total of 8 tests.
Remarks	Up to 180 days	Good bonding strength	None	None	Good bonding strength	None	Types-1 and -4 failures; average bonding strength for all tests – 264 psi (1.8 MPa)

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C6. Bonding Strength of PLS-614 with Dry Concrete CIGMAT CT-3 (Sandwich)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type-4	Type-5	
Dry Concrete	30	××					252
	90	×					279
	180	×					293
Total No. (% Failure)		4 (100%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	Total of 4 tests
Remarks	Up to 180 days	Good bonding strength	None	None	None	None	Type-1 failures; average bonding strength for all tests – 269 psi (1.8 MPa).

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C7. Bonding Strength of PLS-614 with Wet Concrete CIGMAT CT-3 (Sandwich)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type- 2	Type-3	Type-4	Type-5	
Wet Concrete	30	××					292
	90				×		257
	180	×					309
Total No. (% Failure)		3 (75%)	0 (0%)	0 (0%)	1 (25%)	0 (0%)	Total of 4 tests.
Remarks	Up to 180 days	Good bonding strength	None	None	Good bonding strength	None	Type-1 and Type-4 failures; average bonding strength for all tests – 287 psi (2.0 MPa).

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C8. Bonding Strength of PLS-614 with Dry Clay Brick CIGMAT CT-3 (Sandwich)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type-4	Type-5	
Dry Clay Brick	30	××					329
	90	×					331
	180	×					350
Total No. (% Failure)		4 (100%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	Total of 4 tests
Remarks	Up to 180 days	Good bonding strength	None	None	None	None	Type-1 failures; average bonding strength for all tests – 335 psi (2.3 MPa)

Type-1 = Substrate failure
Type-2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

Table C9. Bonding Strength of PLS-614 with Wet Clay Brick CIGMAT CT-3 (Sandwich)

Substrate	Approximate Curing Time (days)	Failure Modes					Average Failure Strength (psi)
		Type-1	Type-2	Type-3	Type-4	Type-5	
Wet Clay Brick	30	××					372
	90	×					338
	180	×					384
Total No. (% Failure)		4 (100%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	Total of 4 tests
Remarks	Up to 180 days	Good bonding strength	None	None	None	None	Type-1 failures; average bonding strength for all tests – 366 psi (2.5 MPa)

Type-1 = Substrate failure
Type- 2 = Coating failure
Type-3 = Bonding failure
Type-4 = Combined substrate and bonding failure
Type-5 = Combined coating and bonding failure

APPENDIX D

Manufacturer Data Sheet for Epoxy Mastic PLS-614

VENDOR DATA SHEET
PHYSICAL PROPERTIES OF COATING

Coating Product Name: Epoxy Mastic PLS-614

Coating Product Vendor Name and Address: **Protective Liner Systems, Inc.**
6691 Tribble Street
Lithonia, Georgia 30058

Coating Type: Epoxy Mastic (PLS-614)

Testing Method	Vendor Results
Tensile strength (ASTM D-63860)	12,400 PSI
Chemical Resistance (ASTM D 543) (3 % H_2 SO_4)	
Water Vapor Transmission (ASTM D 1653/E 1907)	
Flexural Strength (ASTM D 79058T)	13,900 PSI
Hardness- Shore D (ASTM D 2240)	72
Impact Resistance (NCS PS 55-75)	160 in./lbs
Volatile Organic Compounds - VOCs (ASTM D 2832)	None

Worker Safety	Result/Requirement
Flammability Rating	
Known Carcinogenic Content	
Other hazards (corrosive)	

Environmental Characteristics	Result/Requirement
Heavy Metal Content (w/w)	
Leaching of Cured Coating (TCLP)	
Disposal of Cured Coating	

Application Characteristics	Result/Requirement
Primer Requirement	
Number of Coats and Thickness	125 mils. An additional coat can be applied after 2 or 3 hours, but no later than 24 hours.
Minimum Application Temperature	
Minimum Cure Time Before Handling	
Maximum Application Temperature	
Minimum Cure Time before Immersion into Service	Approximately 4 hours at 70ºF
Type of Surface Preparation Before Coating	Remove all oil, grease and foreign materials, including residual laitance. Clean all metals, either to a commercial abrasive blast finish, or by a thorough hand tool cleaning. Galvanized steel and aluminum may require etching. Remove all deteriorated wood to a solid surface.

Vendor Experience	Comments
Length of Time the Coating in Use	
Applicator Training & Qualification Program	
QA/QC Program for Coating/Lining	